AECOM

Year 1 Interim Report: Joint Industry Project Study of Well Treatment, Completion, and Workover Effluents

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Table of Contents

Acronym List vi

Executive Summary viii

1.0 Introduction 1
1.1 Year 1 Interim Report Study Questions 1
1.2 Document Organization 1

2.0 Selection of Structures for Sampling 3
2.1 Statistical Approach 3
2.1.1 Data Screening 3
2.1.2 LHS Input Variables 3
2.2 Selected Structures and Sample Size 3

3.0 TCW Discharge Characteristics 5
3.1 Well Operation Type 5
3.1.1 Well Treatment 5
3.1.2 Well Completion 5
3.1.3 Well Workover 5
3.2 TCW Fluid Composition 5
3.2.1 Brines 6
3.2.2 Chemical Products 7
3.3 Discharge of TCW Effluents to GOM Surface Waters 9
3.3.1 Discharge Configuration 10
3.3.2 Duration of Sampled Discharge 10
3.3.3 Discharge Volume 10
3.4 Treatment of TCW Effluents 12
3.5 Summary 12

4.0 TCW Effluent Composition and Variability 13
4.1 Analytical Laboratories 13
4.2 Laboratory Control Seawater 13
4.2.1 Approach 13
4.2.2 Composition of Laboratory Control Seawater 14
4.3 Effluent Composition at the Critical Effluent Dilution 15
4.3.1 Approach 15
4.3.2 TCW Category I Effluent Composition 16
4.3.3 TCW Category III Effluent Composition 18
4.4 Composition of Undiluted (100%) Effluent 20
4.4.1 Approach 20
4.4.2 TCW Category I Effluent Composition 20
4.4.3 TCW Category III Effluent Composition 24
4.5 Variability in Effluent Composition During a Single Discharge 28
4.5.1 Approach 28
4.5.2 Assessment Results 28
4.6 Summary 29

5.0 Acute Aquatic Toxicity of Discharged TCW Effluents 31

ED_005567_00000011-00002

5.1 Acute 48-h Static Renewal WET Test 31
5.1.1 WET Test Procedures 31
5.1.2 Preparation of Category III Gel Samples 32
5.1.3 Aquatic Toxicity of Category I Effluents 32
5.1.4 Aquatic Toxicity of Category III Effluents 32
5.1.5 Comparison of TCW Category I and III Effluents 33
5.2 Summary 34

6.0 Acute Aquatic Hazard of Added Chemical Products 35
6.1 Hazard Assessment Approach 35
6.1.1 GHS Acute Aquatic Toxicity Classification 35
6.2 TCW Category I Effluents 36
6.2.1 GHS Acute Category 1 36
6.2.2 GHS Acute Category 2 36
6.2.3 GHS Acute Category 3 36
6.3 TCW Category III Effluents 37
6.3.1 GHS Acute Category 1 37
6.3.2 GHS Acute Category 2 37
6.3.3 GHS Acute Category 3 38
6.4 Summary 38

7.0 Potential Causes of Acute Aquatic Toxicity 40
7.1 Patterns in Acute Aquatic Toxicity 40
7.1.1 Approach 40
7.1.2 Ordination Results 40
7.1.3 Summary 43
7.2 Toxicity-Composition Connections 43
7.2.1 Approach 43
7.2.2 Acute Toxic Unit 45
7.2.3 TCW Category I Effluents 45
7.2.4 TCW Category III Effluents 47
7.3 Acute Toxicity Screening at the CD 48
7.3.1 Acute Toxicity Screening Approach 48
7.3.2 Substances Potentially Contributing to Toxicity at the CD 49
7.4 Summary 50

8.0 Recommendations for Year 2 JIP Study Activities 52
8.1 Sample Collection Schedule 52
8.2 Sample Size 52
8.3 Laboratory Analysis 52
8.4 Reporting 52

9.0 Conclusions 53

10.0 References 55

Text Tables

Table 1	LHS Input Variables used to Select Structures for Sampling
Table 2	TCW Effluent Sample Codes
Table 3	Categories of TCW Fluids Sampled in Year 1
Table 4	Descriptive Statistics for Analytical Parameters Measured in Laboratory Control Seawater
Table 5	Descriptive Statistics for Analytical Parameters Measured in TCW Category I Effluents at the CD
Table 6	Descriptive Statistics for Analytical Parameters Measured in TCW Category III Effluents at the CD
Table 7	Raw Data and Descriptive Statistics for Directly Measured Parameters in Undiluted TCW Category I Effluents
Table 8	Raw Data and Descriptive Statistics for Estimated Dissolved Ions in Undiluted TCW Category I Effluents
Table 9	Raw Data and Descriptive Statistics for Estimated TSS and TOC in Undiluted TCW Category I Effluents
Table 10	Raw Data and Descriptive Statistics for Directly Measured Parameters in Undiluted TCW Category III Effluents
Table 11	Raw Data and Descriptive Statistics for Estimated Dissolved Ions in TCW Category III Effluents
Table 12	Raw Estimates and Descriptive Statistics for Estimated TSS and TOC in TCW Category III Effluents
Table 13	Change in Analytical Parameters at Different Times in the Discharge Expressed as a Ratio
Table 14	Acute 48-h whole effluent toxicity of TCW Category I Effluents
Table 15	Acute 48-h whole effluent toxicity of TCW Category III Effluents
Table 16	Inland silverside minnow and Mysid Acute WET Test Endpoint by Cluster Analysis Grouping
Table 17	Spearman rank-order Correlation of TUa and Estimated Dissolved Ion Concentration
Table 18	Tier 1 Acute Toxicity Screening at the Critical Effluent Dilution: Exceedances
Table 19	Tier 2 Refinements of the Acute Toxicity Screening at the Critical Effluent Dilution

Appendix Tables

Table A1	TCW Effluent Sample Area, Block, and API Well No.
Table A2	TCW Effluent Discharge Characteristics
Table A3	Substances Potentially Present in Brines and Chemical Products by TCW Effluent Sample

Table A4 Laboratory Analytical Parameters

Table A5 Laboratory Analytical Results for Laboratory Control Seawater and TCW Effluent Samples

Table A6 GHS Acute Aquatic Toxicity Classification of Chemical Products in TCW Category I Effluents

Table A7 GHS Acute Aquatic Toxicity Classification of Chemical Products in TCW Category III Effluents

Table A8 Acute Aquatic Life Ecological Screening Values

Text Figures

Figure 1 Boxplot of Discharge Duration (Hours) for TCW Category I and Category III Effluents

Figure 2 Boxplot of Discharge Volume (bbl) for TCW Category I and Category III Effluents

Figure 3 Boxplots for Specific Gravity, Salinity, Alkalinity, and pH of Undiluted TCW Category I Effluents

Figure 4 Bar Charts of Estimated Dissolved Cations and Anions in Undiluted TCW Category I Effluents

Figure 5 Bar Chart of Estimated TOC in Undiluted TCW Category I Effluents

Figure 6 Bar Chart of Estimated TSS in Undiluted TCW Category I Effluents

Figure 7 Boxplots for Specific gravity, Salinity, Alkalinity, and pH of Undiluted TCW Category III Effluents

Figure 8 Bar Charts of Estimated Dissolved Cations and Anions in Undiluted TCW Category III Effluents

Figure 9 Bar Chart of Estimated TOC in Undiluted TCW Category III Effluents

Figure 10 Bar Chart of Estimated TSS in Undiluted TCW Category III Effluents

Figure 11 Boxplots for Mysid and Inland Silverside Minnow 48-H LC50s for TCW Category I and III Effluents

Figure 12 Acute Toxic Unit (TUa) by TCW Effluent Sample

Figure 13 Cluster Analysis Dendrogram of the Inland silverside minnow and Mysid Acute Toxic Unit WET Test Endpoints

Figure 14 Polynomial Regression of the Mysid and Inland Silverside Minnow TUa with Dissolved Calcium in TCW Category I Effluents

Figure 15 Polynomial Regression of the Mysid and Inland Silverside Minnow TUa with Dissolved Calcium in TCW Category III Effluents

Figure 16 Polynomial Regression of the Mysid and Inland silverside minnow TUa with DOC and TOC in TCW Category III effluents

Appendix Figures

Figure A1	TCW Discharge Sample Locations
Figure A2	Photographs of TCW Category III Gels
Figure A3	Mixing Apparatus for Category III Gel Samples

Appendices

Appendix A	JIP Study Participant Survey Questionnaire Form
Appendix B	Raw Output for Latin Hypercube Sampling Evaluation
Appendix C	Supporting Documentation for Statistical Analyses
Appendix D	Water Accommodated Fraction (WAF) Aquatic Toxicity Test Procedure and Results
Appendix E	ProUCL Documentation

Acronym List

Acronym	Explanation
%	Percent
API	American Petroleum Institute
bbl	Barrel
bbl/day	Barrel per Day
CAS	Chemical Abstracts Service
CD	Critical Effluent Dilution
cm	Centimeter
CMC	Criterion Maximum Concentration
CV	Coefficient of Variation
DDAC	Didecyldimethylammonium Chloride
DOC	Dissolved Organic Carbon
DQO	Data Quality Objective
EEUSA	Environmental Enterprises USA, Inc.
EMTL	Element Materials Technology Lafayette
EPC	Exposure Point Concentration
ESV	Ecological Screening Value
ft.	Feet
GAC	Granular Activated Carbon
GHS	Globally Harmonized System
GOM	Gulf of Mexico
GP	General NPDES Permit
JIP	Joint Industry Project
L	Liter
LC25	25 Percent Lethal Concentration
LC50	50 Percent Median Lethal Concentration
LCSW	Laboratory Control Seawater
L(E)C50	Median Lethal (or Effects) Concentration
LHS	Latin Hypercube Sampling
LOEC	Lowest Observed Effect Concentration
LOEL	Lowest Observed Effect Loading
m	Meter
meq/L	Milliequivalents Per Liter
mg/L	Milligrams per Liter
NOEC	No Observed Effect Concentration
NPDES	National Pollutant Discharge Elimination System
OOC	Offshore Operators Committee
p	Probability
PAH	Polycyclic Aromatic Hydrocarbon
PP	Priority Pollutant
ppt	Parts per Thousand
QAC	Quaternary Ammonium Compound
RL	Reporting Limit
RPM	Revolutions per Minute
SDS	Safety Data Sheet
SU	Standard Unit
TAC	Test Acceptability Criteria
TBP	Tributyl Phosphate
TCW	Treatment, Completion, and Workover
TDS	Total Dissolved Solids
TOC	Total Organic Carbon
TQ	Toxicity Quotient
TSS	Total Suspended Solids

Acronym	Explanation
TTPC	Tributyl Tetradecyl Phosphonium Chloride
TUa	Acute Toxic Unit
UCL	Upper Confidence Limit
USEPA	U.S. Environmental Protection Agency
VOC	Volatile Organic Compound
WAF	Water Accommodated Fraction
WET	Whole Effluent Toxicity
w/w	Weight by Weight

Executive Summary

This report presents the Year 1 results of a two-year joint industry project (JIP) study of well treatment, completion, and workover (TCW) effluents discharged to Gulf of Mexico (GOM) surface waters. The JIP study was commissioned to enable JIP study participants to meet their National Pollutant Discharge Elimination System (NPDES) General Permit (GP) requirements for characteristic assessments of TCW fluids under the Industry-Wide Study Alternative. The Year 1 evaluations characterized TCW discharges and assessed the potential for TCW effluent characteristics to contribute to acute whole effluent toxicity. The Year 1 data provide a better understanding of TCW effluent characteristics, their aquatic toxicity, and substances that potentially contribute to this toxicity. A summary of Year 1 JIP study findings is provided below:

- **What are the characteristics of discharged TCW effluents?** A total of 23 TCW effluent samples were collected across 19 discharge structures between November 2019 and May 2020. Of the four categories of TCW fluids identified during planning for this study, only TCW Category I completion brine-based fluids, and TCW Category III workover and treatment fluids were discharged during Year 1. Neither TCW Category II nor TCW Category IV fluids were discharged to the GOM during the study period. TCW Category I and TCW Category III fluids are comprised of chloride or bromide brines and may contain chemical products that contain organic substances.
- **How are TCW effluents discharged?** Discharges of TCW effluents were made through a pipe or hose and averaged 2.5 hours in duration.
- **What is the typical chemical composition of discharged TCW effluents?** TCW Category I and III effluents at the critical effluent dilution (CD) are comprised of metals, cations and anions, and organics. Concentrations of some substances were highly variable, reflecting changes in TCW fluid composition needed to achieve well operational objectives. Some variability in effluent chemical composition was observed when evaluated over the duration of a discharge.
- **What is the acute toxicity of discharged TCW effluents?** Acute 48-hour (48-h) whole effluent toxicity (WET) testing was conducted with *Menidia beryllina* (Inland silverside minnow) and *Americamysis bahia* (Mysid). TCW effluents exhibited a wide range of acute toxicities. The arithmetic mean LC50 for Inland silverside minnow was 12% effluent, with LC50s ranging from 0.6% to >50% effluent. The Mysid was more sensitive to TCW effluents than was the Inland silverside minnow. The arithmetic mean LC50 Mysid was 9% effluent, with LC50s ranging from 0.54% to 35% effluent. A subset of TCW Category III effluents were gels. These were the most toxic effluents collected.
- **What are the aquatic hazard characteristics of chemical products?** Safety Data Sheets (SDS) for a minority of chemical products used in TCW fluids provided aquatic hazard information. For these products, an aquatic hazard assessment was conducted consistent with the United Nations (2019) guidance *A Guide to The Globally Harmonized System of Classification and Labeling of Chemicals (GHS) 8th Edition*. Among the chemical products whose SDS presented GHS classifications, there were products in each of the three GHS acute aquatic toxicity categories: GHS Category 1 – Very toxic; GHS Category 2 – Toxic; and GHS Category 3 – Harmful. For the majority of the chemical

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products used in TCW fluids, no GHS data was presented in SDS and no assessment of hazard was conducted.

- **What is contributing to the observed acute aquatic toxicity at the end of pipe?** The cation Ca^{2+} appeared to contribute to Mysid toxicity in some TCW Category I effluents, whereas there was no association of Ca^{2+} with toxicity to the Inland silverside minnow. In TCW Category III effluents, dissolved organic carbon (DOC) and total organic carbon (TOC) appear to influence Inland silverside minnow toxicity. The toxicity may be at least partially attributable to organic substances in chemical products.

  Based on the aquatic hazard characterization, organic substances in chemical products that may contribute to the observed acute toxicity were quaternary ammonium compounds (QACs), tributyl phosphate (TBP), and tributyl tetradecyl phosphonium chloride (TTPC). These additives are used in products that provide the following chemical functionalities: cationic surfactants, lytic biocides, corrosion inhibitors, and non-emulsifiers.

- **What substances may potentially contribute to acute aquatic toxicity in the receiving water at the edge of the mixing zone?** An acute toxicity screening evaluation conducted at the CD identified dissolved arsenic and total copper as having the potential to contribute to aquatic toxicity. The concentrations of these substances were equal to conservative aquatic ecological screening values (ESVs). This suggests there is some potential for acute aquatic toxicity at the edge of the mixing zone. Exceedances of aquatic ESVs for these metals are primarily associated with TCW Category III effluents.

The Year 2 sampling will occur from February to April 2021. The final report will be submitted to USEPA Region 4 and Region 6 in October 2021 consistent with the study plan. The recommendation for Year 2 is to continue with the approach in Year 1. Year 2 sampling will continue to use the sample mixing technique adopted as a USEPA-approved study plan change in Year 1 to conduct WET testing of gel effluents. Additional analyses of laboratory control seawater (LCSW) will be made to better define background concentrations. As discussed in the study plan, any refinements to the Year 2 JIP study activities will be discussed with USEPA Regions 4 and 6 before they are implemented.

1.0 Introduction

This interim report presents the Year 1 results of a two-year joint industry project (JIP) study of well treatment, completion, and workover (TCW) effluents discharged to Gulf of Mexico (GOM) surface waters. The JIP study was commissioned to enable JIP study participants to meet their National Pollutant Discharge Elimination System (NPDES) General Permit (GP) requirements for characteristic assessments of TCW fluids under the Industry-Wide Study Alternative. The objectives of the JIP study are to characterize the chemical composition and acute aquatic toxicity of TCW effluents, and their potential to cause acute aquatic toxicity to GOM aquatic biota. The information in this report provides U.S. Environmental Protection Agency (USEPA) Region 4 and Region 6 with an update on Year 1 results and informs Year 2 JIP study activities consistent with the August 19, 2019 study plan.

1.1 Year 1 Interim Report Study Questions

To achieve JIP study objectives and inform the Year 2 JIP study design, this report addresses the following study questions:

- What substances are currently used in TCW fluids? What are their general aquatic hazard characteristics?
- How are TCW effluents typically handled and their discharge to GOM surface waters managed?
- What are the estimated concentrations of substances in GOM surface waters at the critical effluent dilution (CD), i.e., the concentration predicted to exist in the effluent plume at the edge of the 100-meter (m) mixing zone?
- What is the typical chemical composition of discharged TCW effluents? How variable is the chemical composition of a discharge?
- How toxic are TCW effluents towards marine biota?
- Can general toxicity-composition connections be made?
- What substances could potentially be associated with acute aquatic toxicity at the CD?
- Can sampling and analysis in Year 2 be adjusted to better address JIP study objectives?

1.2 Document Organization

The interim report sections are presented below:

- **Section 2.0** Selection of Structures for Sampling.
- **Section 3.0** TCW Effluent Discharge Characteristics.
- **Section 4.0** TCW Effluent Composition and Variability.
- **Section 5.0** Acute Aquatic Toxicity of Discharged TCW Effluents.
- **Section 6.0** Acute Aquatic Hazard of Chemical Products.
- **Section 7.0** Potential Causes of Acute Aquatic Toxicity.
- **Section 8.0** Recommendations for Year 2 JIP Study Activities.

- **Section 9.0** Conclusions.
- **Section 10.0** References.

Essential figures and tables are incorporated into the body of the report. Supplemental figures and lengthy tables are presented at the end of the report and are referenced where needed in the text. This report also includes the following appendices:

- **Appendix A** JIP Study Participant Survey Questionnaire Form
- **Appendix B** Raw Output for Latin Hypercube Sampling Evaluation
- **Appendix C** Supporting Documentation for Statistical Analyses
- **Appendix D** Water Accommodated Fraction (WAF) Aquatic Toxicity Test Procedure and Results
- **Appendix E** ProUCL Documentation

2.0 Selection of Structures for Sampling

This section describes the approach used to select structures for sampling. The GP requirements for TCW fluid characteristic assessments (USEPA, 2017) under the Industry-Wide Study Alternative specify examination of a "*statistical[ly] valid number of samples of wells in the Western and Central [for USEPA Region 6] areas of the GOM*". A total of 19 offshore platforms and vessels ("structures") were sampled within the GOM central planning area. The structures were selected from a database of 95 planned discharges generated by JIP study participants using a survey questionnaire. An example questionnaire is provided in **Appendix A**. Structure selection was objective and intended to yield representative data that characterize the likely range of discharged TCW effluents within the GOM study area.

2.1 Statistical Approach

Samples discussed in this report were collected between November 2019 and May 2020. In 2019, a total of three structures were identified by JIP study participants and were sampled. In 2020, a larger number of structures were available and statistical sub-sampling consistent with the USEPA-approved study plan was warranted. The statistical approach is *n*-dimensional Latin hypercube sampling (LHS) (McKay et al., 1979). LHS is a stratified random procedure that provides an efficient way of sampling multiple input variables (Minasny and McBratney, 2006). Raw LHS output for the selected variables are provided in **Appendix B**.

2.1.1 Data Screening

Each of the 95 planned TCW effluent discharges was screened for consistency with JIP study data quality objectives (DQOs). Discharges were eliminated from consideration for sampling if the TCW effluents were comingled with produced water or if the available information had insufficient detail to conduct the LHS analysis. The screened discharges were carried forward for LHS evaluation.

2.1.2 LHS Input Variables

A total of 16 input variables deemed important for generating representative data were selected from the JIP study participant database (**Table 1**). The input variables fall into the following categories: geographical, TCW fluid category, type of chemical products, and type of TCW effluent treatment. Input variables were either continuous or discrete.

2.2 Selected Structures and Sample Size

The LHS algorithm selected 34 structures for evaluation in 2020. Field data collection was paused in June 2020 after sampling 19 structures over a period of 7 months to develop protocols to address difficult-to-analyze samples. A total of 24 TCW effluent samples were collected from the 19 structures; each sample was assigned a randomized sample code (**Table 2**). The lease area, block, and American Petroleum Institute (API) well number for the 24 TCW effluent samples are provided in **Table A1**. Individual sample locations are shown in **Figure A1**.

<table>
<tr><th colspan="4">Table 1. LHS Input Variables used to Select Structures for Sampling</th></tr>
<tr><th>Input Variable Type</th><th>Input Variable</th><th>Discrete or Continuous?</th><th>Rationale for Selection</th></tr>
<tr><td rowspan="2">Geographical</td><td>Block No.</td><td>Discrete</td><td rowspan="2">Spatial aspect; position within the study area.</td></tr>
<tr><td>Water Column Depth</td><td>Continuous</td></tr>
<tr><td rowspan="4">TCW Fluid Category</td><td>Category I</td><td rowspan="13">Discrete:
"Absent" = 0;
"Present" = 1</td><td rowspan="13">Can influence whole discharge toxicity and chemical makeup of discharge.</td></tr>
<tr><td>Category II</td></tr>
<tr><td>Category III</td></tr>
<tr><td>Category IV</td></tr>
<tr><td rowspan="5">Chemical products</td><td>Corrosion Inhibitors</td></tr>
<tr><td>Non-emulsifiers</td></tr>
<tr><td>Surfactants</td></tr>
<tr><td>Defoamers</td></tr>
<tr><td>Biocides</td></tr>
<tr><td rowspan="4">TCW Effluent Treatment</td><td>No Treatment or Tank Storage</td></tr>
<tr><td>Tank Storage</td></tr>
<tr><td>Filtration</td></tr>
<tr><td>Other Treatment, e.g. polishing step</td></tr>
</table>

Table 2. TCW Effluent Sample Codes.
HV63
JK70
RD67
RU61
XP62
NY50
LC54
YO64
AU71
FP89
ZG57
GQ67
YU91
LX98
IS88
RU72
IH80*
BT52
SH87
EP57 (begin); TR84 (end)
RC74 (begin); OD76 (middle); and TF74 (end)
Notes: * After collecting sample IH80, the Operator determined that the sample was not discharged to GOM surface water. This sample was therefore not representative of TCW discharges.

3.0 TCW Discharge Characteristics

This section addresses the following JIP study questions:

- *What substances are currently used in TCW fluids?* This question is addressed by identifying the categories of TCW fluids discharged to GOM surface waters and describing the type and general composition of TCW brines and chemical products that make up the fluids.
- *How are TCW effluents typically handled and their discharge to GOM surface waters managed?* This question is addressed by describing the effluent discharge configuration; effluent discharge duration, volume, and rate; and treatment of effluents before discharge to surface water, where applicable.

3.1 Well Operation Type

Well operation types represented by the sampled structures were well treatment, completion, and workover. Detailed information associated with each operation e.g., TCW fluid category and discharge characteristics is provided in **Table A2**.

3.1.1 Well Treatment

Of the 24 TCW effluent samples collected, 9 were associated with well treatment operations such as hydraulic fracturing, chemical treatment, wellbore cleanout and acidizing.

3.1.2 Well Completion

A total of 11 TCW effluent samples were associated with completion operations. Completion operations involve using solids-free brines to complete a well and facilitate final operations before production. The brine's density is selected to provide sufficient hydrostatic pressure to control the well. Completion fluids may also contain polymers and other additives.

3.1.3 Well Workover

A total of four TCW effluent samples were identified by JIP study participants as being from workover operations. Workover refers to the process of performing major maintenance or remedial treatments on a well or to set packers. Workover fluids are typically brines that are free of solids and that will not adversely affect either the reservoir fluids or the formation.

3.2 TCW Fluid Composition

There are four categories of TCW fluids (TCW Categories I-IV). The choice of fluid category depends on the type of well operation. A description of each TCW fluid category is provided below in **Table 3**.

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Table 3. Categories of TCW Fluids and Fluids Sampled in Year 1		
TCW Category	**Description**	**No. of Sampled Discharges**
I	Typically clear, brine-based fluids use to treat, complete, or workover a well. May be comprised of fresh water or saltwater brines of appropriate density for well control. May contain some chemical products.	9
II	Organic (acetic and formic acids) and inorganic acids (hydrochloric and hydrofluoric) and/or blends of each.	0 (Not discharged to the GOM during Year 1)
III	Category III fluids typically use a Category I fluid as the base component. One or more additional chemical products are added to achieve desired properties: • Small amounts of polymers, e.g., guar, are used to give the fluid viscosity. • Cross-linkers, e.g., boron are used to create a “Jell-O” like fluid consistency. Supporting additives used to improve the cross-link function or improve the performance of the fluid include: buffers to maintain favorable fluid pH to stabilize the cross-link; surfactants to improve reservoir wettability and fluid recovery; and breakers that ensure that the cross-link breaks as designed.	15
IV	• Can be classified as a treatment, completion or workover fluid depending on how it is used. • The use of hydrocarbon-based fluids in TCW fluids is infrequent and normally limited to the removal of waxes and asphaltenes from the wellbore and/or sand face. • Some hydrocarbons can be gelled to act as fracturing fluids, but that is only when water-based fluids are damaging to the reservoir. This is not common in the offshore environment. • Gelled hydrocarbons may also be used as packer fluids to control convective heat transfer in wells that have high bottom hole temperatures or high flow rates that create a high-temperature environment that could damage ancillary equipment. • Base oils can be used to perform negative pressure testing for regulatory compliance.	0 (Not discharged to the GOM)

During the sampling conducted to date, only TCW Category I and III fluids were used and then discharged. TCW Category I and TCW Category III fluids are comprised of brines and chemical products. Individual anions and cations, and other substances potentially present in chemical products are presented by TCW effluent sample in **Table A3**.

3.2.1 Brines

Brines form the base for TCW fluids:

- **TCW Category I**: Category I fluids are used in completion operations. The two classes of brines observed during the study are chloride brines: calcium chloride ($CaCl_2$), sodium chloride (NaCl), and potassium chloride (KCl); and bromide brines: calcium bromide ($CaBr_2$) and sodium bromide (NaBr).

- **TCW Category III**: Category III fluids are used in workover, treatment and fracturing operations. In addition to a chloride or bromide brine base, Category III fluids contain additional components that provide needed functional properties.

3.2.2 Chemical Products

Chemical products beyond inorganic salts are added to TCW fluids to support well operations and protect piping and associated infrastructure. The types of chemical products used varied with the type of operation. The chemical functionalities provided by the chemical products used include:

- Defoamers.
- Friction reducers.
- Scale inhibitors.
- Iron control.
- Oxygen scavengers.
- Mutual solvents.
- Acid inhibitors.
- Corrosion inhibitors.
- Viscosifiers.
- Clay stabilizers.
- Surfactants.
- Non-emulsifiers.
- Breakers.
- pH control.
- Cross-linkers.
- Gel stabilizers.
- Proppants.
- Biocides.
- Well cleaners and spacers.

Product Safety Data Sheets (SDSs) were consulted for information on chemical composition of the chemical products used. A summary of the dominant functionalities provided by chemical products is provided below by TCW category. Trade names of chemical products are not provided. Instead, chemical additive codes based on chemical functionality are used to identify chemical additives used in the study. SDSs sometimes only list chemicals by functionality, e.g., "surfactant" rather than by chemical name; this limitation is reflected in the following discussion.

- **TCW Category I**: Completion chemical products are used to clean wells after drilling, to control them while they are being perforated, and to make them operational when essential equipment such as packers and tubing are added (Boehm, Turton, Raval, Caudle, French, Rabalais, Spies, and Johnson, 2001). In some instances, no chemical products other than inorganic salts were present in

TCW Category I fluids. When present, chemical products included biocides, acid treatments, scale inhibitors, non-emulsifiers, de-foamers, viscosifiers, and pH control agents as described below:

- Biocides are used to control microbiological growth in piping and other infrastructure. The chemical product "Biocide 1" was an example of a biocide present in TCW Category I effluents and contains the aldehyde glutaraldehyde.
- Acid treatments: In one instance, a treatment with acetic and hydrochloric acids was observed for a completion operation with a Category I brine. Acetic acid is used in high-temperature wells, typically in conjunction with hydrochloric acid.
- Scale inhibitors: Seawater often reacts with the formation water to produce inorganic scales or deposits of barium or calcium salts that must be controlled with scale inhibitors. One anti-scaling product that was present in Category I effluents is "Scale inhibitor 2", which is composed of the inorganic salt sodium molybdate and the organic solvent ethylene glycol.
- Non-emulsifiers: Surfactants are sometimes added to Category I fluids to prevent the formation of emulsions between completion brines containing calcium, e.g., $CaBr_2$, $CaCl_2$ and crude oil. For example, the product "Non-emulsifier 1" contains proprietary quaternary ammonium compounds (QACs) that are cationic surfactants.
- De-foamers: Unwanted foams can result when using surfactants. The chemical product "Defoamer 1" was one product used in TCW Category I fluids. The phosphate ester tributyl phosphate (TBP) (30 - 60 percent weight by weight [w/w%]) is a key component of this product.
- Viscosifers: The product "Viscosifier 1" was used in a TCW Category I fluid in support of a workover operation to viscosify low weight brines.
- pH Control: pH control agents can be used to facilitate the control of bacteria or to raise the pH of acidic fluids. Addition of sodium hydroxide is used to control pH. A commercial pH control product used in TCW Category I samples for this study was "pH Control 3".

- **TCW Category III**: Category III fluids used in treatment operations contain chemical products added to a brine base to achieve specific functional properties. Chemical products are present in all Category III effluents. Synthetic mud casing scrubbers, clay control chemicals, polymers, cross-linkers, and proppant beads were used in various samples collected during the study. Other types of chemical products include biocides, corrosion inhibitors, oxygen scavengers, scale inhibitors, well casing cleaner, de-foamers, pH control agents, non-emulsifiers, and solvents. Details of these chemical products are provided below:
    - Polymers and Cross-linkers (Gels) are only used in Category III fluids. Polymers such as guar gum and xanthum gum are used to form gels. Cross-linkers, e.g., ammonium chloride, potassium hydroxide and borate salts, also create a gel-like fluid consistency and were present in TCW fluids. Gel samples were YO64, YU91, and OD76. Representative

photographs of gels, including a sample with embedded proppant beads, are provided in **Figure A2**.

- Biocides are used to control bacterial consumption of polymers present in TCW Category III gels, and to minimize microbiological growth in piping and other infrastructure. The chemical products "Biocide 4" and "Biocide 2" are examples of biocide products used in this study. Common components of these biocides include glutaraldehyde and didecyldimethylammonium chloride (DDAC). Biocides are often, but not always, used in hydraulic fracturing fluid treatments (Kahrilas, Blotevogel, Stewart, and Borch, 2015) and other fluids containing polymers.
- Corrosion inhibitors and oxygen scavengers: Corrosion protection is necessary to ensure safe drilling operations. "Corrosion Inhibitor 1" is one product that was used as a corrosion inhibitor in TCW Category III fluids and consists of reducing agents, alcohols, and acids. Oxygen scavengers remove soluble oxygen from water-based drilling and completion fluids. "Oxygen Scavenger 1" is another product used for corrosion control; this product is a liquid oxygen scavenger containing the inorganic reducing agent ammonium bisulfite.
- Scale inhibitors: One anti-scaling product commonly used during the study is "Scale Inhibitor 2", which is comprised of ethylene glycol and sodium molybdate.
- Well casing cleaner: TCW effluent samples containing well casing cleaner were observed. One well-cleaner used in study samples was "Well Cleaner 1", which is comprised of surfactants and solvents used in fluid displacement and cleanup operations.

  "Fluid additive 1" was another well cleaner product used during the study that contains surfactants, solvents, and water-wetting agents. In one instance, a soap pill was used in a workover operation to scour and remove debris from the well hole. An example of a cleaning pill used during the study was comprised of a mixture of NaBr and $CaBr_2$ brines and a well cleaning product ("Surfactant 2").
- De-foamers: "Defoamer 2" was one product used as an antifoam agent; it is composed of the neutral organics kerosene, naphthalene, and ethylbenzene. "Defoamer 3" was another product used as a de-foamer in Category III fluids.
- pH Control: pH control consists of the addition of sodium hydroxide. An example of a pH control product used was "pH Control 3".
- Non-emulsifiers: A cationic polymer in solution ("Non-emulsifier 2") was used in TCW Category III discharges as a non-emulsifier.
- Solvents were also present in Category III effluents. "Solvent 1" contains acetic acid and the neutral organics xylene and 2-butoxyethanol.

3.3 Discharge of TCW Effluents to GOM Surface Waters

This section describes discharge configuration, duration, and volume of TCW Category I and TCW Category III effluents. This information illustrates how the discharge of TCW effluents to GOM surface waters is managed.

3.3.1 Discharge Configuration

The selected structures were situated in deep waters. Arithmetic mean water column depth at the discharge structures was 1,616 meters (m), with a maximum of 2,913 m. The difference between the seafloor and the end-of-pipe averaged 1,548 m, with a range of 46 - 2,901 m. The discharge of TCW effluents typically occurred through a pipe on the structure ranging in diameter from 8 - 46 centimeters (cm.). The depth of the end-of-pipe ranged from 12 m below to 27 m above the water surface. There were exceptions to these characteristics. In one instance, effluents were discharged through a 41 cm diameter pipe that was mounted flush with the hull of a vessel that houses the structure. Also, two structures discharged TCW effluents through a submerged Tideflex Diffuser "Duckbill" system. A submerged diffuser improves hydrodynamic mixing of the TCW effluent with GOM surface waters.

3.3.2 Duration of Sampled Discharge

TCW effluent discharges were intermittent and of short duration. The discharge of TCW Category III effluents occurred over a shorter duration than TCW Category I discharges (**Figure 1**):

- **TCW Category I effluents**: Discharges averaged 5 hours, with a range of 0.03 - 24 hours. The longest discharge duration was associated with a long-term completion (flow-back) operation. TCW effluents in that case were only discharged over a 24-hour period at the beginning of the 31-day flow-back period.
- **TCW Category III effluents**: Category III discharges averaged 1 hour, with a range of 0.05 - 3.38 hours.

3.3.3 Discharge Volume

Typical effluent discharge volumes (barrel or “bbl”) depend on the type of well and the specific operation being performed. The median volume of discharged TCW Category I effluents was lower than that reported for TCW Category III (**Figure 2**). The volume of TCW Category I discharges averaged 868 bbls, with a range of 10 - 2,534 bbls. The volume of TCW Category III discharges averaged 520 bbls with a range of 30 - 1,577 bbls.

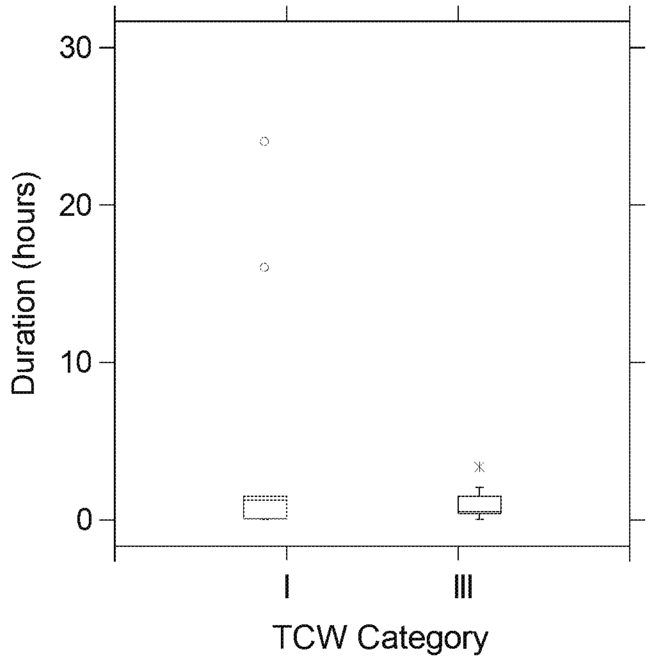

Figure 1. Boxplot of discharge duration (hours) for TCW Category I (n=9) and TCW Category III effluents (n=14). The center line marks the median. Box edges are at the first and third quartiles. Whiskers show the range of observed values that fall within 1.5x of the interquartile range of the box edges. Outliers (*) and extreme outliers (°) are shown. Additional details on boxplots are provided in **Appendix C**.

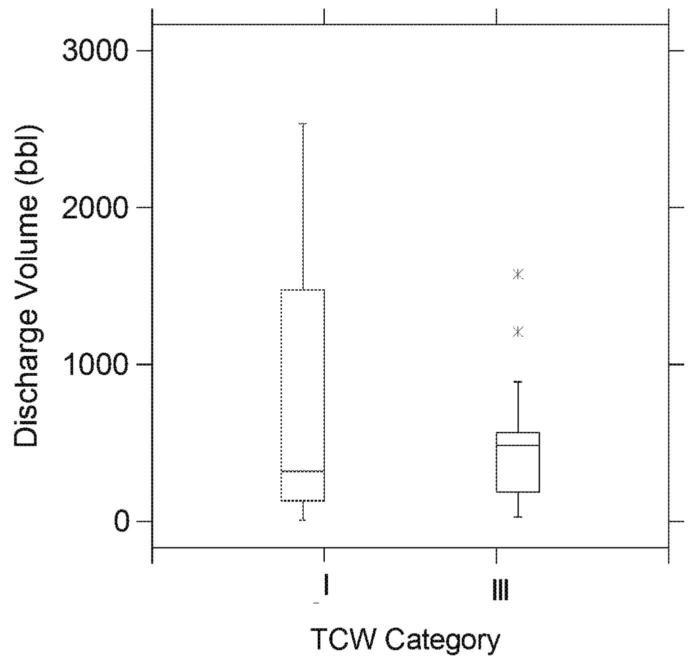

Figure 2. Boxplot of discharge volume (bbl) for TCW Category I (n=9) and TCW Category III effluents (n=14). The center line marks the median. Box edges are at the first and third quartiles. Whiskers show the range of observed values that fall within 1.5x of the interquartile range of the box edges. Outliers (*) are shown. Additional details on boxplots are provided in **Appendix C**.

3.4 Treatment of TCW Effluents

Treatment of TCW effluent varies across discharge structures (**Table A2**). In some instances, treatment was used to neutralize pH before the effluents are discharge. More advanced treatment of TCW effluents was observed at discharge structures No.10 and No.18, where the well operation was completion (flow-back). The treatment package for these structures included surge tanks, a weir box, solids filters, absorption media, and granulated activated carbon (GAC) vessels. GAC can be used to polish discharges for residual organics and dissolved oil removal via carbon adsorption (Igwe, Saadi, and Ngene, 2013).

3.5 Summary

Section 3.0 identifies the characteristics of TCW effluent discharges. Based on the information provided, the JIP study questions identified at the beginning of Section 3.0 can be addressed as follows:

- **What substances are currently used in TCW fluids?** TCW fluids are comprised of brines and chemical products. Chloride and bromide brines were used during the study period. Chemical products are largely comprised of organic substances. Chemical products were not always present in TCW Category I fluids but were always used in TCW Category III fluids.
- **How are TCW discharges typically handled and their discharge to GOM surface waters managed?** TCW effluents were discharged through a pipe or hose in most cases. In two instances, the discharge occurred through a submerged diffuser. TCW discharges to GOM surface waters occur intermittently and average 2.5 hours in duration. TCW Category I discharges lasted longer than TCW Category III discharges. The discharge volume of TCW Category I discharges was greater than that reported for TCW Category III discharges. The range of TCW discharge volumes was 10 - 2,534 bbls. In some cases, there was end-of-pipe treatment of TCW effluents.

4.0 TCW Effluent Composition and Variability

This section describes TCW effluent composition and variability, and addresses the following JIP study questions:

- *What are the concentrations of substances in GOM surface waters at the critical effluent dilution, i.e., the concentration predicted to exist in the effluent plume at the edge of the 100-meter (m) mixing zone?*
- *What is the typical chemical composition of discharged TCW effluents?*
- *How variable are the concentrations of substances over the duration of the discharge?*

4.1 Analytical Laboratories

Three analytical laboratories were used to support the chemical analysis of TCW effluent samples. Environmental Enterprises USA, Inc. (EEUSA; Slidell, LA) conducted the analysis of water quality parameters on samples of undiluted (100%) effluent, prepared samples for chemical analysis at the critical effluent dilution (CD) with laboratory control seawater (LCSW) and shipped the prepared samples to Element Materials Technology Lafayette (Element; Lafayette, LA). Element conducted the analysis of selected analytical parameters. Element subcontracted ALS Environmental (ALS; Kelso, WA) to conduct total and dissolved mercury (Hg) analysis.

4.2 Laboratory Control Seawater

The concentrations of 59 analytical parameters were measured in two samples of synthetic LCSW used to prepare the TCW effluent samples at the CD. Laboratory chemical analysis was conducted to understand how LCSW potentially contributes to TCW effluent quality. The coefficient of variation (CV) was used to characterize variability in chemical composition. Laboratory analytical parameters are provided in **Table A4**; tabulated analytical data for LCSW are presented in **Table A5**.

4.2.1 Approach

The synthetic LCSW was prepared by EEUSA with hw-MARINEMIX + Bio-elements, Crystal Sea Marinemix Bioassay Laboratory Formula sea salts (80:20), and deionized water. This mixture was adjusted to a salinity of 25 parts per thousand (ppt). Laboratory analytical parameters measured in LCSW are summarized below:

- **Water quality parameters**: DOC and TOC; alkalinity, total; alkalinity, bicarbonate (estimated as 1.22 * total alkalinity); hardness, total (as $CaCO_3$); TSS; nitrogen, ammonia (as N); and chemical oxygen demand (COD). The parameters DOC, TOC, and COD were used to indicate the presence of organic substances.
- **Metals**: 11 total and dissolved Priority Pollutant (PP) metals, basic cations, and basic anions were analyzed. Mercury (HG was analyzed
- **Organics**: The 16 PP polycyclic aromatic hydrocarbons (PAHs).

The CV (%) was used as a descriptive measure of variability for analytical parameters. The CV is the ratio of the standard deviation (σ) to the arithmetic mean ($\bar{x}$). The ratio was converted to a percentage. A CV of 100% indicates that σ and $\bar{x}$ are equal. A CV greater than 100% indicates that the parameter of interest was highly variable among the samples tested.

4.2.2 Composition of Laboratory Control Seawater

Descriptive statistics for LCSW are provided below in **Table 4**. Detailed results are presented by sample in **Table A5**.

Table 4. Descriptive Statistics for Analytical Parameters Measured in Synthetic Laboratory Control Seawater.

Parameter	Count	Detects	Freq. of Detection	Max. (mg/L)	Arith. Mean (mg/L)	Std. Dev. (mg/L)	CV
Water Quality Parameters (Total)							
Hardness (as CaCO3)	2	2	100%	4,430	4,290	198	5%
Alkalinity, Total (As CaCO3)	2	2	100%	93	74	27	36%
HCO_3^- (Estimated as 1.22 * Total Alk.)	2	2	100%	113	90	32	36%
Total Suspended Solids (Residue, Non-Filterable)	2	0	0%	--	--	--	--
Nitrogen, Ammonia (As N)[1]	2	0	0%	--	--	--	--
Chemical Oxygen Demand	2	0	0%	--	--	--	--
Organic Carbon, Total	2	0	0%	--	--	--	--
Sulfide	2	1	50%	0.03	0.03	0	28%
Specific Gravity	2	0	0%	--	--	--	--
Water Quality Parameters (Dissolved)							
Total Dissolved Solids (Residue, Filterable)	2	2	100%	24,400	22,350	2,899	13%
Dissolved Organic Carbon	2	0	0%	--	--	--	--
Metals (Total)							
As	2	0	0%	--	--	--	--
Ba	2	1	50%	0.02	0.06	0	90%
Cd	2	2	100%	0.01	0.01	0.01	104%
Ca	2	2	100%	273	267	8	3%
Cr	2	0	0%	--	--	--	--
Cu	2	1	50%	0.02	0.02	0	39%
Pb	2	0	0%	--	--	--	--
Mg	2	2	100%	910	879	44	5%
Hg	2	2	100%	0.000004	0.000002	0.000002	88%
Ni	2	0	0%	--	--	--	--
K	2	2	100%	283	282	2	1%
Se	2	2	100%	0.31	0.22	0.12	56%
Na	2	2	100%	6,630	6,595	49	1%
Tl	2	0	0%	--	--	--	--
Zn	2	1	50%	0.01	0.06	0.06	111%
Metals (Dissolved)							
As	2	0	0%	--	--	--	--
Ba	2	1	50%	0.02	0.06	0.05	88%
Cd	2	1	50%	0.002	0.006	0.006	90%
Ca	2	2	100%	259	258	2	1%
Cr	2	0	0%	--	--	--	--
Cu	2	1	50%	0.01	0.03	0.03	83%
Pb	2	0	0%	--	--	--	--
Mg	2	2	100%	848	843	8	1%
Hg	2	1	50%	0.0000011	0.0000008	0.0000004	53%
Ni	2	0	0%	--	--	--	--
K	2	2	100%	278	261	25	10%
Se	2	1	50%	0.15	0.17	0.17	100%
Na	2	2	100%	6,790	6,745	64	1%
Tl	2	2	100%	0.12	0.07	0.08	126%
Zn	2	0	0%	--	--	--	--
Inorganic Anions (Total)							
Br	2	2	100%	38	38	0.07	0%
Cl	2	2	100%	13,700	13,350	495	4%
SO_4^{2-}	2	2	100%	2,070	1,950	170	9%
Polycyclic Aromatic Hydrocarbons (PAHs)							
PAHs	2	0	0%	--	--	--	--

The composition of LCSW and composition variabilities are summarized below

- **Substances not detected** above the laboratory RL in LCSW were: TSS, nitrogen, ammonia, COD, TOC, DOC, total metals (As, Cr, Pb, Ni, and Tl); dissolved metals (As, Cr, Pb, Ni, and Zn); and 16 PAHs.
- **Substances with 100% detection frequency** were: hardness; alkalinity (total and bicarbonate); TDS; total metals (Cd, Ca, Mg, Hg, K, Se, Na); dissolved metals (Ca, Mg, K, Na, Tl); and inorganic anions (Br^-, Cl^-, and sulfate [$SO_4^{(2-)}$]). Total Hg was typically detected near the method reporting limit (0.0000005 mg/L).
- **Variability in analytical parameters**: Detected analytical parameters with a CV greater than 100% included Cd (104%), total Zn (111%), and dissolved Tl (126%).

4.3 Effluent Composition at the Critical Effluent Dilution

The concentrations of 59 analytical parameters were also measured at the CD for TCW effluent samples. As discussed above, the samples were prepared with LCSW. The CV was used to characterize variability in chemical composition at the CD. Tabulated analytical data measured in TCW effluent samples at the CD are presented in **Table A5**.

4.3.1 Approach

Laboratory analytical parameters were measured at the critical effluent dilution concentration (CD) consistent with the study plan:

- **Estimation of the CD**: Estimated CDs were provided to EEUSA so that samples for chemical analysis could be prepared. The CD was estimated by scaling the observed discharge volume (barrels [bbl]) to a daily discharge rate (barrels per day [bbl/day]) using discharge durations provided by JIP Study participants. This information was combined with discharge pipe diameter (inches) and the depth difference between end-of-pipe and seafloor (meters) to estimate the CD. Consistent with the study plan, CDs were obtained from the produced water critical effluent dilution tables provided in Appendix D of the Region 6 GP (USEPA, 2017). All the samples collected in Year 1 were from discharges occurring in Region 6 waters.
- **Laboratory analytical parameters**: The same suite of analytical parameters evaluated for LCSW was evaluated in the TCW effluent samples. Due to the nature of the discharge and mixing of toxicity test samples during sample preparation, the loss of VOCs through volatilization may occur. Hence, VOCs were not analyzed in TCW effluent samples.
- **Samples not analyzed**: Samples IH80 and BT52 were not subjected to chemical analysis. Sample IH80 formed two phases when mixed with laboratory control seawater and was not submitted for analysis. It was later determined that this fluid was never discharged. Insufficient sample volume was collected in the field to analyze BT52.
- **Coefficient of variation (CV)**: The CV (%) was used as a descriptive measure of variability for analytical parameters as described above for LCSW. Elevated variability in TCW effluents can potentially result from operation type, type of brine and chemical products, and other factors, e.g., formation rock type.

4.3.2 TCW Category I Effluent Composition

TCW Category I effluents at the CD (average CD = 0.44% effluent) were comprised of inorganics and organic chemical products. Descriptive statistics are provided below in **Table 5**. Detailed results are presented by sample in **Table A5**. The composition of Category I effluents and composition variabilities are summarized below:

- **Substances not detected** above the laboratory RL were nitrogen, ammonia; As (diss.); total/dissolved metals (Cr, Pb, and Ni); and 16 PAHs.
- **Substances with 100% detection frequency** were: hardness; alkalinity (total and bicarbonate); TDS; total metals (Ca^{2+}, Mg^{2+}, Hg, K, and Na), dissolved metals (Ca^{2+}, Mg^{2+}, K, and Na); and inorganic anions (Br^-, Cl^-, and sulfate [$SO_4^{(2-)}$]). Total Hg was typically detected near the method reporting limit (0.0000005 mg/L).
- **Variability in analytical parameters**: Detected analytical parameters with a CV greater than 100% included Br (249%), DOC (187%), TOC (185%), thallium (Tl) (121%), and copper (Cu) (107%). The variability in TOC and DOC of Category I effluents may reflect the presence or absence of chemical products. For example, TOC and DOC concentrations below the RL were associated with effluent samples that did not have any chemical products present (HV63 and XP62). TOC and DOC were also not detected in effluents where GAC treatment was present (ZG57, EP57, and TR84). The variability of cation and anion concentrations, other than that of Br^-, was low.
- **Maximum concentrations**: The maximum concentration of Br^- (2,630 mg/L) was observed at sample RU61 (completion operation with a $CaBr_2$ brine and acetic/HCl acid treatment). Maximum TOC (406 mg/L) and DOC (385 mg/L) were also observed at RU61. The elevated DOC and TOC for RU61 may be associated with acetic acid. The maximum concentration of Tl (0.008 mg/L), which is above the laboratory RL (0.006 mg/L), was observed in two samples (RD67 and RU61). The maximum concentration of copper (0.046 mg/L) was observed for EP57; this sample was collected at the beginning of a completion operation flow-back. Cu was also detected in the LCSW (arithmetic mean = 0.023 mg/L).

Table 5. Descriptive Statistics for Analytical Parameters Measured in TCW Category I Effluents at the CD.							
Parameter	**Count**	**Detects**	**Freq. of Detection**	**Max. (mg/L)**	**Arith. Mean (mg/L)**	**Std. Dev. (mg/L)**	**CV**
Water Quality Parameters (Total)							
Hardness (as CaCO3)	9	9	100%	5,810	4,461	1,461	33%
Alkalinity, Total (As CaCO3)	9	9	100%	98	81	8	10%
HCO_3^- (Estimated as 1.22 * Total Alk.)	9	9	100%	119	98	10	10%
Total Suspended Solids (Residue, Non-Filterable)	9	7	78%	19	12	5	39%
Nitrogen, Ammonia (As N)	9	0	0%	--	--	--	--
Chemical Oxygen Demand	9	1	11%	1,420	1,420	--	--
Organic Carbon, Total	9	4	44%	406	108	199	185%
Sulfide	9	6	67%	0.03	0.03	0.004	17%
Specific Gravity	9	9	100%	1.5	1.2	0.1	12%
Water Quality Parameters (Dissolved)							
Total Dissolved Solids (Residue, Filterable)	9	9	100%	29,700	24,567	2,832	12%
Dissolved Organic Carbon	9	4	44%	385	101	189	187%
Metals (Total)							
As	9	1	11%	0.1	0.1	--	--
Ba	9	3	33%	0.1	0.05	0.03	52%
Cd	9	4	44%	0.01	0.004	0.004	100%
Ca	9	9	100%	834	489	243	50%
Cr	9	0	0%	--	--	--	--
Cu	9	6	67%	0.05	0.03	0.02	78%
Pb	9	0	0%	--	--	--	--
Mg	9	9	100%	935	884	43	5%
Hg	9	9	100%	0.000002	0.000001	0.0000005	34%
Ni	9	0	0%	--	--	--	--
K	9	9	100%	381	308	43	14%
Se	9	7	78%	0.5	0.3	0.1	44%
Na	9	9	100%	7,690	7,061	359	5%
Tl	9	2	22%	0.008	0.008	0	0%
Zn	9	4	44%	0.1	0.1	0.1	57%
Metals (Dissolved)							
As	9	0	0%	--	--	--	--
Ba	9	3	33%	0.1	0.1	0.1	83%
Cd	9	3	33%	0.002	0.002	0.0001	4%
Ca	9	9	100%	808	471	233	49%
Cr	9	0	0%	--	--	--	--
Cu	9	4	44%	0.05	0.02	0.02	107%
Pb	9	0	0%	--	--	--	--
Mg	9	9	100%	901	853	26	3%
Hg	9	7	78%	0.000002	0.000001	0.0000004	42%
Ni	9	0	0%	--	--	--	--
K	9	9	100%	373	301	30	10%
Se	9	7	78%	0.4	0.2	0.1	40%
Na	9	9	100%	7,260	6,924	192	3%
Tl	9	2	22%	0.1	0.05	0.1	121%
Zn	9	3	33%	0.2	0.1	0.1	75%
Inorganic Anions (Total)							
Br	9	9	100%	2,630	344	857	249%
Cl	9	9	100%	15,700	14,167	903	6%
SO_4^{2-}	9	9	100%	2,140	1,950	171	9%
Polycyclic Aromatic Hydrocarbons (PAHs)							
PAHs	9	0	0%	--	--	--	--

4.3.3 TCW Category III Effluent Composition

Category III effluents at the CD (average CD = 0.37% effluent) were comprised of inorganics (cations and anions) and organics from chemical products, e.g., well cleaner. The CVs for TOC and DOC were lower than reported for Category I effluents because organic chemical products were present in all Category III effluent samples. Descriptive statistics are provided below in **Table 6**; detailed results are presented in **Table A5**. The composition of Category III effluents and composition variabilities are summarized below:

- **Substances not detected** above the RL were total and dissolved metals (Cr, Pb, and Ni); and 16 PAHs.
- **Substances with 100% detection frequency** were hardness; alkalinity (total/bicarbonate); TDS; total metals (Ca^{2+}, Mg^{2+}, Hg, K, and Na); dissolved metals (Ca^{2+}, Mg^{2+}, K, and Na); and inorganic anions (Br^-, Cl^-, and $SO_4^{(2-)}$).
- **Variability in analytical parameters**: Detected analytical parameters with a CV greater than 100% included: Br^- (314%), total Ca^{2+} (132%), dissolved As (128%), dissolved Tl and DOC (127%), dissolved Ca^{2+} (119%), total Tl (114%), TOC (107%), and dissolved cadmium (102%). Maximum concentrations of these parameters are described below.
- **Maximum concentrations**: The maximum concentrations of Br (8,850 mg/L) and total Ca^{2+} (2,370 mg/L) were reported for effluent sample TF74 (a treatment operation/fracturing job reverse-out). The sample was collected at the end of the treatment operation and consisted of a $CaCl_2$ brine. The maximum concentration of dissolved As (0.288 mg/L) was reported for RU72 (treatment operation). RU72 was a sample of a Category III KCl brine “frac-pack” and proppant beads were present in the sample. Maximum detected concentrations of COD (960 mg/L), TOC (70.3 mg/L), and DOC (126 mg/L) were reported for sample YO64. This Category III gel sample was collected from a treatment operation. Chemical products containing organics were present in this sample.

Table 6. Descriptive Statistics for Analytical Parameters Measured in TCW Category III Effluents at the CD.							
Parameter	Count	Detects	Freq. of Detection	Max. (mg/L)	Arith. Mean (mg/L)	Std. Dev. (mg/L)	CV
Water Quality Parameters (Total)							
Hardness (as CaCO3)	13	13	100%	9,720	4,615	1,604	35%
Alkalinity, Total (As CaCO3)	13	13	100%	105	79	13	16%
HCO_3^- (Estimated as 1.22 * Total Alk.)	13	13	100%	128	95	17	18%
Total Suspended Solids (Residue, Non-Filterable)	13	12	92%	77	23	18	78%
Nitrogen, Ammonia (As N)	13	1	8%	1	0.5	--	--
Chemical Oxygen Demand	13	2	15%	960	770	269	35%
Organic Carbon, Total	13	7	54%	70	23	24	107%
Sulfide	13	5	38%	0.03	0.02	0.003	15%
Specific Gravity	13	9	69%	1.7	1	0.2	21%
Water Quality Parameters (Dissolved)							
Total Dissolved Solids (Residue, Filterable)	13	13	100%	39,400	26,215	4,856	19%
Dissolved Organic Carbon	13	6	46%	126	36	46	127%
Metals (Total)							
As	13	1	8%	0.2	0.2	--	--
Ba	13	2	15%	0.1	0.1	0.1	96%
Cd	13	3	23%	0.01	0.01	0.004	53%
Ca	13	13	100%	2,370	442	582	132%
Cr	13	0	0%	--	--	--	--
Cu	13	7	54%	0.1	0.04	0.01	31%
Pb	13	0	0%	--	--	--	--
Mg	13	13	100%	993	853	96	11%
Hg	13	13	100%	0.00001	0.000002	0.000002	88%
Ni	13	0	0%	--	--	--	--
K	13	13	100%	499	339	83	25%
Se	13	8	62%	0.5	0.3	0.1	33%
Na	13	13	100%	7,640	6,692	711	11%
Tl	13	2	15%	0.1	0.1	0.1	114%
Zn	13	3	23%	0.2	0.1	0.1	82%
Metals (Dissolved)							
As	13	2	15%	0.3	0.2	0.2	128%
Ba	13	2	15%	0.1	0.1	0.1	97%
Cd	13	2	15%	0.01	0.01	0.01	102%
Ca	13	13	100%	2,140	432	516	119%
Cr	13	0	0%	--	--	--	--
Cu	13	1	8%	0.01	--	--	--
Pb	13	0	0%	--	--	--	--
Mg	13	13	100%	1,030	872	78	9%
Hg	13	12	92%	0.000002	0.000001	0.000001	45%
Ni	13	0	0%	--	--	--	--
K	13	13	100%	504	337	75	22%
Se	13	10	77%	0.5	0.3	0.1	26%
Na	13	13	100%	8,310	6,969	683	10%
Tl	13	2	15%	0.1	0.1	0.1	127%
Zn	13	1	8%	0.4	--	--	--
Inorganic Anions (Total)							
Br	13	13	100%	8,850	775	2,430	314%
Cl	13	13	100%	14,500	13,808	373	3%
SO_4^{2-}	13	13	100%	2,230	1,935	135	7%
Polycyclic Aromatic Hydrocarbons (PAHs)							
PAHs	13	0	0%	--	--	--	--

4.4 Composition of Undiluted (100%) Effluent

The evaluations presented in this subsection address undiluted TCW effluents, i.e., before mixing with GOM surface waters or LCSW. Chemical composition was determined by directly measuring 4 analytical parameters and estimating the concentrations of 10 analytical parameters, using the results of analyses of samples diluted to the CD.

4.4.1 Approach

Laboratory analytical parameters were either directly measured or estimated to assess the composition of undiluted effluents:

- **Directly measured parameters**: Analytical parameters directly measured in undiluted effluent (only for aqueous, non-gel samples) are specific gravity (@4˚C; salinity (parts per thousand [ppt]); alkalinity, as calcium carbonate ($CaCO_3$); and pH (standard units [S.U.]).
- **Estimated parameters**: Parameters estimated in undiluted effluent are cations (Na, Mg, K, and Ca); anions (HCO_3^-, Cl^-, and Br^-); TOC; and TSS. Details of the estimation approach are provided below:
  - Dissolved cations and anions: The concentrations of cations and anions in undiluted effluent (C_{TCW}) were estimated where: C_{TCW} = (C_{sample}-C_{LCSW}*(1-CD/100))/(CD/100) and: C_{sample} = concentration at the CD, and C_{LCSW} = concentration in laboratory control seawater. Estimates of C_{TCW} are not reliable, however, unless C_{sample} > C_{LCSW}; these estimates were not used. Mass to volume ratios, e.g., milligrams per liter (mg/L) do not accurately represent the exposure of a WET test organism to an individual ion. Instead, organisms are exposed to individual ions within a salt molecule. Hence, cation and anion concentrations were converted from mg/L to milliequivalents per liter (meq/L).
  - TOC and TSS: The concentrations of TOC (milligrams per liter or "mg/L") and TSS (mg/L) were estimated where: C_{TCW100} = C_{TCWCD}*(100/C_{sample}). Non-detect values were converted to ½ of the laboratory RL.

4.4.2 TCW Category I Effluent Composition

Undiluted Category I effluents were denser than seawater due to their elevated salinity and can be alkaline, with effluent reaching a pH of 10.0 S.U. Details of the effluent composition evaluations are presented below:

- **Directly measured parameters**: Undiluted TCW Category I effluents exhibited a specific gravity range of 1.02 to 1.45, are highly saline (arithmetic mean of 222 ppt), exhibit an alkalinity range of 20 to >400 mg/L, and are somewhat acidic to alkaline (pH range 6.6 to 10) (**Figure 3**). Raw data and descriptive statistics for substances are provided below in **Table 7**.

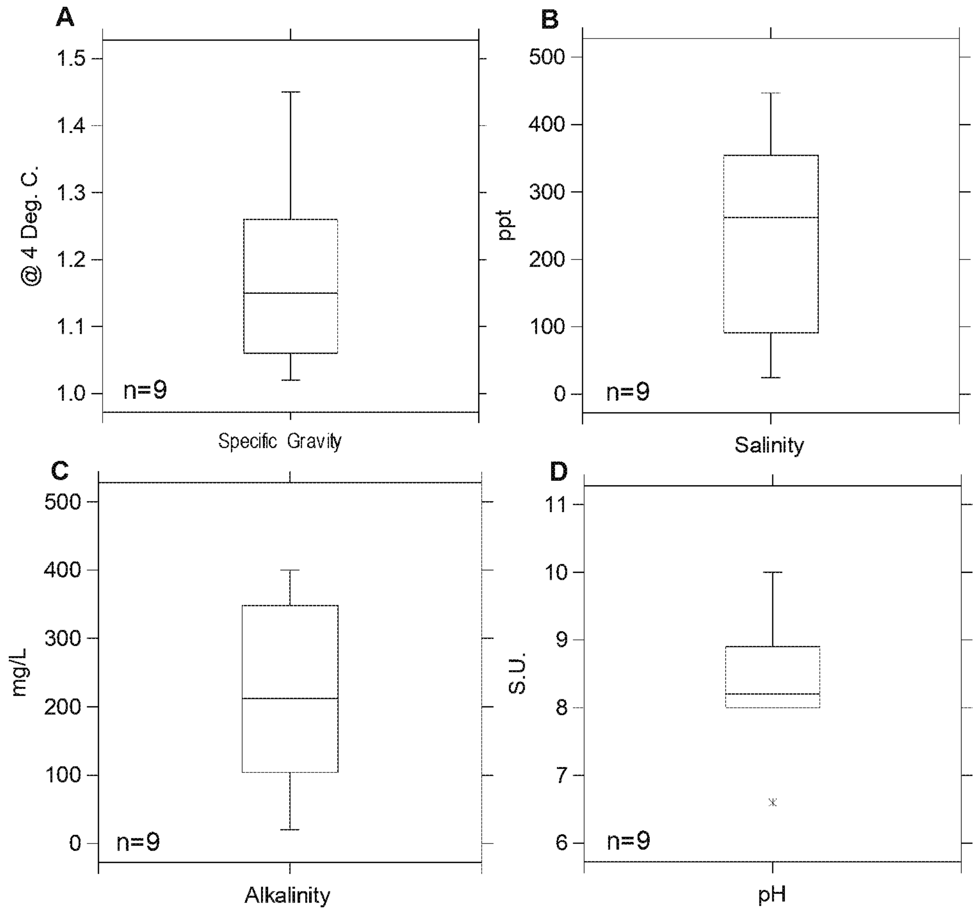

Figure 3. Boxplots for specific gravity, salinity, alkalinity, and pH of undiluted TCW Category I effluents (n=9). The center line marks the median. Box edges are at the first and third quartiles. Whiskers show the range of observed values that fall within 1.5x of the interquartile range of the box edges. Outliers (*) are shown. Additional details on boxplots are provided in **Appendix C**.

Table 7. Raw Data and Descriptive Statistics for Directly Measured Parameters in Undiluted TCW Category I Effluents.

Sample	Specific Gravity	Alkalinity, as CaCO3 (mg/L)	Salinity (ppt)	pH (S.U.)
HV63	1.26	104	358	8.3
RD67	1.24	>400	354	10.0
RU61	1.45	136	295	6.6
XP62	1.30	>400	447	9.8
LC54	1.06	72	103	8.0
AU71	1.15	20	262	8.0
ZG57	1.02	348	24.5	8.9
EP57	1.05	212	64.1	8.2
TR84	1.06	288	91	8.1
n	9	9	9	9
Arith. Mean	1.18	220	222	--
Min.	1.02	20	24.5	6.6
Max.	1.45	>400	447	10.0

- **Estimated dissolved cations and anions**: TCW effluent sample XP62 (a $CaCl_2$ brine) exhibited the highest combined Na and Ca^{2+} milliequivalents (11,739 meq/L) (**Figure 4A**). In contrast, ZG57 exhibited cation/anion milliequivalents that are lower than observed in the LCSW, with the exceptions of K (143 meq/L) and HCO_3^- (27.2 meq/L) (**Table 8**). This is likely a consequence of the low salinity reported for ZG57. The maximum value for K (782 meq/L) was reported for EP57. The maximum for Cl^- is 15,226 meq/L estimated for sample AU71 (NaCl brine) (**Figure 4B**). The maximum milliequivalent for Br (5,900 meq/L) was reported for sample RU61 ($CaBr_2$ brine).

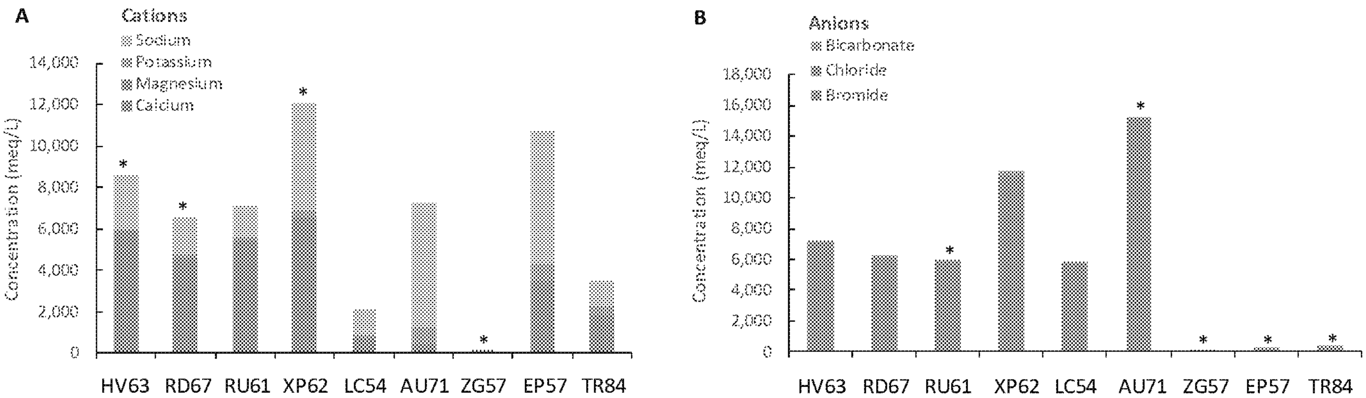

Figure 4. Bar charts of estimated dissolved cations and anions in undiluted TCW Category I effluents. A " * " indicates that the concentration of one or more substances in the TCW effluent sample was less than observed in the laboratory control seawater, and the estimated values were negative. These data are not included in the bars shown.

A table of estimated concentrations and descriptive statistics for detected substances are provided below in **Table 8**.

Table 8. Raw Data and Descriptive Statistics for Estimated Dissolved Ions in Undiluted TCW Category I Effluents.

Parameter	Mg^{2+}	K^+	Na^+	Ca^{2+}	Br^-	Cl^-	HCO_3^-
Units	meq/L	meq/L	meq/L	meq/L	meq/L	meq/L	meq/L
HV63	$C_{sample}<C_{LCSW}$	108	2,615	5,848	2	7,118	7
RD67	$C_{sample}<C_{LCSW}$	164	1,697	4,633	205	5,968	17
RU61	421	130	1,519	5,017	5,900	$C_{sample}<C_{LCSW}$	15
XP62	$C_{sample}<C_{LCSW}$	336	5,213	6,526	28	11,528	146
LC54	165	112	1,250	535	23	5,688	39
AU71	375	744	6,035	135	21	15,226	$C_{sample}<C_{LCSW}$
ZG57	$C_{sample}<C_{LCSW}$	143	$C_{sample}<C_{LCSW}$	$C_{sample}<C_{LCSW}$	$C_{sample}<C_{LCSW}$	$C_{sample}<C_{LCSW}$	27
EP57	3,079	782	6,407	497	136	$C_{sample}<C_{LCSW}$	111
TR84	1,461	476	1,225	334	236	$C_{sample}<C_{LCSW}$	61
n	5	9	8	8	8	5	8
Arith. mean	1,100	333	3,245	2,941	819	9,106	53
Min.	165	108	1,225	135	2	5,688	7
Max.	3,079	782	6,407	6,526	5,900	15,226	146

- **Estimated TOC (mg/L)**: Arithmetic mean estimated TOC for Category I effluent samples is 8,894 mg/L. This result is driven by RU61 (TOC = 73,818 mg/L) (**Figure 5; Table 9**). Without RU61, arithmetic mean TOC for Category I effluents was 778 mg/L. TCW effluent sample HV63 had the lowest estimated TOC (227 mg/L); TOC for the remaining samples ranged from 344 to 1,974 mg/L.

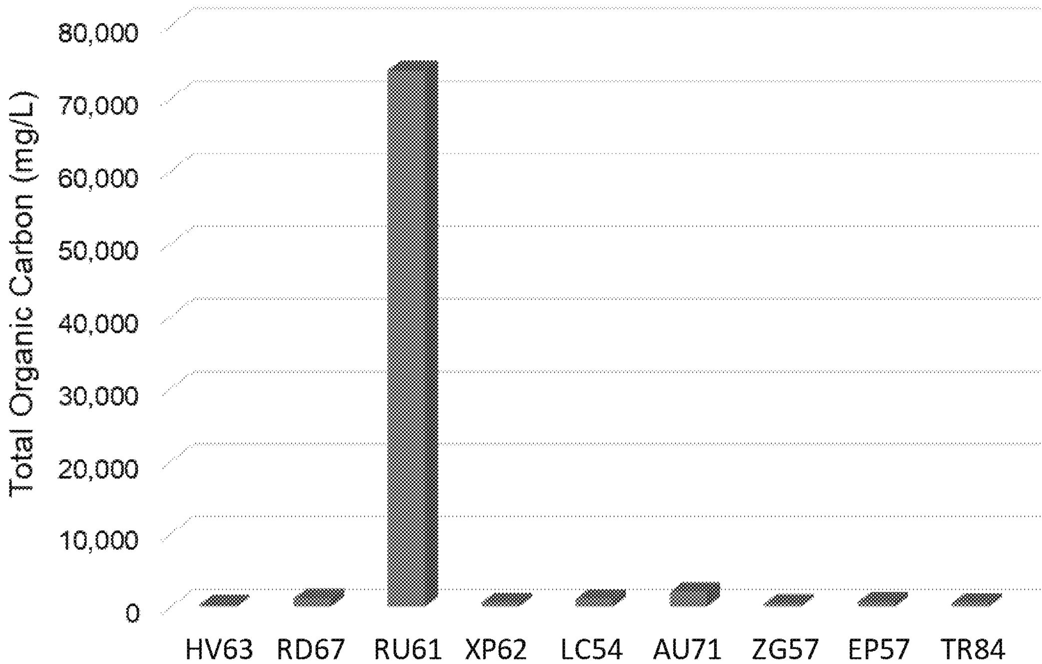

Figure 5. Bar chart of estimated TOC in undiluted TCW Category I effluents.

- **Estimated TSS (mg/L)**: The highest estimated concentration of TSS was reported for TR84 (8,095 mg/L), and the lowest concentration was reported for LC54 (200 mg/L) (**Figure 6**). TSS for the remaining samples ranged from 859 to 7,625 mg/L.

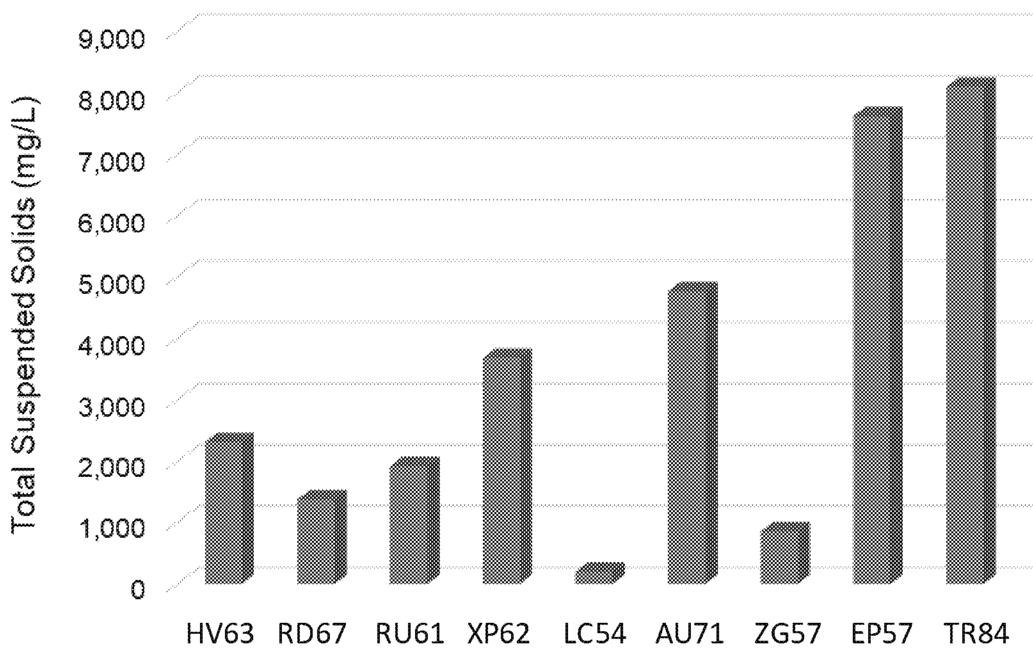

Figure 6. Bar chart of estimated TSS in undiluted TCW Category I effluents.

Table 9. Raw Data and Descriptive Statistics for Estimated TSS and TOC in Undiluted TCW Category I Effluents.		
Parameter	**TSS**	**TOC**
Units	mg/L	mg/L
HV63	2,318	227
RD67	1375	1,083
RU61	1,927	73,818
XP62	3,684	526
LC54	200	968
AU71	4,769	1,974
ZG57	859	344
EP57	7,625	625
TR84	8,095	476
n	9	9
Arith. mean	3,428	8,894
Min.	200	227
Max.	8,095	73,818

4.4.3 TCW Category III Effluent Composition

Category III effluents are less saline than Category I effluents. Category III effluents also possess higher TOC and TSS than Category I effluents. Details of the Category III effluent composition evaluations are presented below:

- **Directly measured parameters**: Category III effluents exhibited a specific gravity range of 1.01 to 1.66, an arithmetic mean salinity of 133 ppt, an alkalinity range of 80 to >400 mg/L and have a circumneutral to alkaline pH (range 7.6 to 11.3) (**Figure 7**). Raw data and descriptive statistics are provided below in **Table 10**.

Table 10. Raw Data and Descriptive Statistics for Directly Measured Parameters in Undiluted TCW Category III Effluents. Greater than (>) values for hardness were defaulted to 400 mg/L when calculating arithmetic mean.				
Sample	**Specific Gravity**	**Alkalinity, as CaCO3 (mg/L)**	**Salinity (ppt)**	**pH (S.U.)**
JK70	1.03	148	57.8	7.7
NY50	1.12	292	175	7.9
YO64	Not available; gel	Not available; gel	Not available; gel	Not available; gel
FP89	1.04	120	64.5	7.6
GQ67	1.49	>400	390	9.1
YU91	Not available; gel	Not available; gel	Not available; gel	Not available; gel
LX98	1.01	80	23.7	7.7
IS88	1.02	144	34.6	7.6
RU72	1.04	>400	58.5	9.8
BT52	Insufficient Sample Volume	>400	58.5	9.9
SH87	1.05	356	80	9.9
RC74	1.01	>400	72	11.3
OD76	Not available; gel	Not available; gel	Not available; gel	Not available; gel
TF74	1.66	200	451	7.9
n	10	11	11	11
Arith. mean	1.15	267	133	--
Min.	1.01	80	23.7	7.6
Max.	1.66	>400	451	11.3

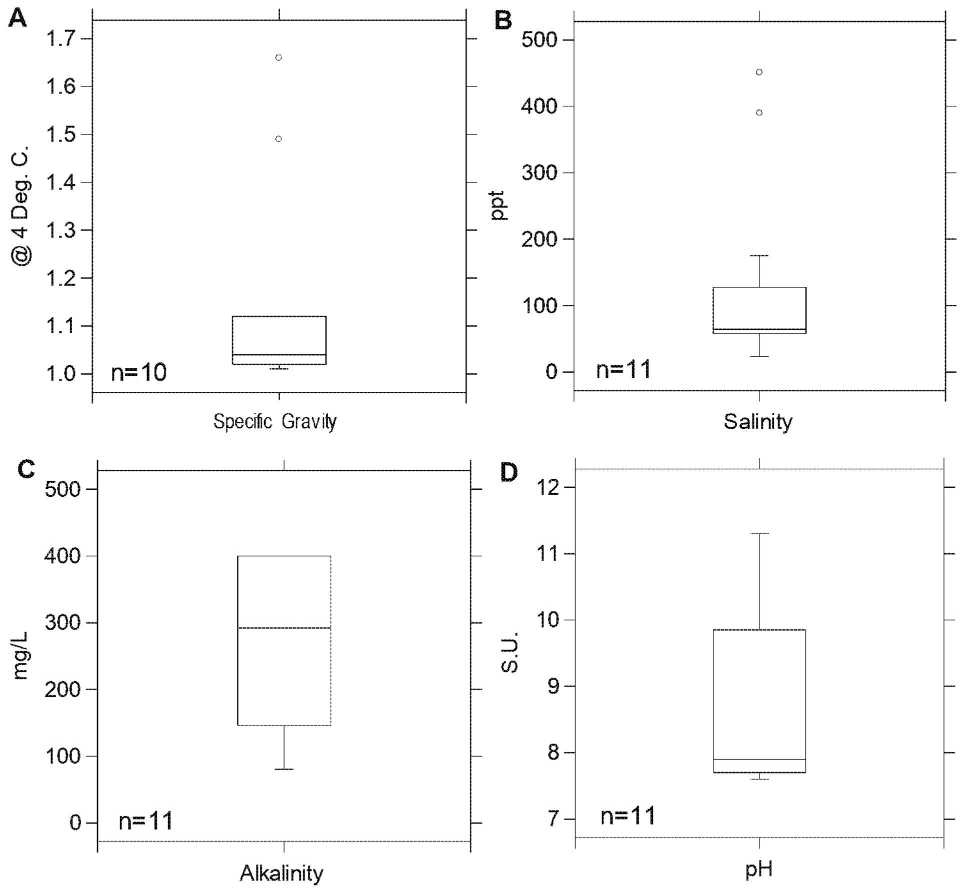

Figure 7. Boxplots for specific gravity, salinity, alkalinity, and pH of undiluted TCW Category III effluents. The center line marks the median. Box edges are at the first and third quartiles. Whiskers show the range of observed values that fall within 1.5x of the interquartile range of the box edges. Extreme outliers (°) are shown. Additional details on boxplots are provided in **Appendix C**.

- **Estimated dissolved cations/anions**: Na is the dominant cation in Category III effluents (**Figure 8A**). Ca^{2+} is highest at sample TF74, the maximum for Cl^-, K and Mg were observed for RC74 (**Figure 8B**). Br^- was highest for sample TF74, and the maximum for HCO_3^- was reported for RU72 (175 meq/L).

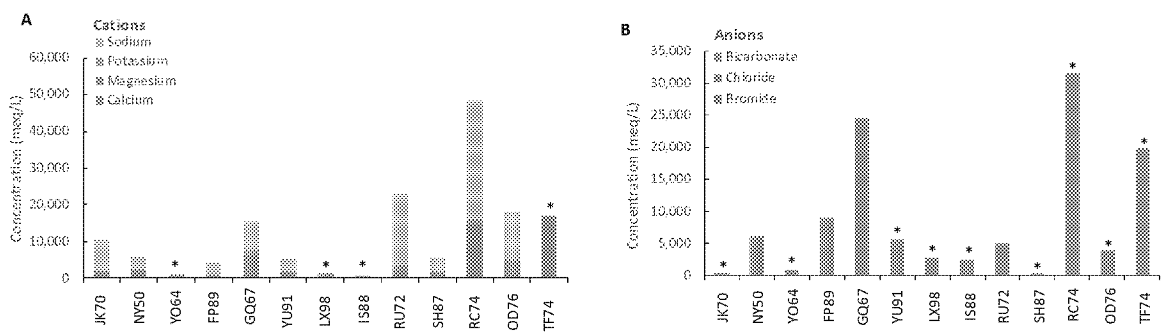

Figure 8. Bar charts of estimated dissolved cations and anions in undiluted TCW Category III effluents. A " * " indicates that the concentration of one or more substances in the TCW effluent sample was less than observed in the laboratory control seawater, and the estimated values are negative. These data are not included in the bars.

A table of estimated concentrations and descriptive statistics for detected substances are provided below in **Table 11**.

Table 11. Raw Data and Descriptive Statistics for Estimated Dissolved Ions in TCW Category III Effluents.

Parameter	Mg^{2+}	K^{+}	Na^{+}	Ca^{2+}	Br^{-}	Cl^{-}	HCO_3^{-}
Units	meq/L	meq/L	meq/L	meq/L	meq/L	meq/L	meq/L
JK70	1,895	89	8,041	353	1	$C_{sample}<C_{LCSW}$	$C_{sample}<C_{LCSW}$
NY50	502	344	3,025	1,840	169	5,810	110
YO64	101	253	$C_{sample}<C_{LCSW}$	647	724	$C_{sample}<C_{LCSW}$	85
FP89	291	712	3,025	45	1	8,707	21
GQ67	851	1,017	7,902	5,738	5,663	18,739	76
YU91	1,344	259	3,209	312	53	5,545	$C_{sample}<C_{LCSW}$
LX98	$C_{sample}<C_{LCSW}$	1,119	$C_{sample}<C_{LCSW}$	$C_{sample}<C_{LCSW}$	5	2,647	$C_{sample}<C_{LCSW}$
IS88	$C_{sample}<C_{LCSW}$	733	$C_{sample}<C_{LCSW}$	$C_{sample}<C_{LCSW}$	3	2,333	$C_{sample}<C_{LCSW}$
RU72	2,001	1,211	19,194	381	40	4,693	175
SH87	1,379	390	3,521	157	25	$C_{sample}<C_{LCSW}$	39
RC74	13,156	1,260	32,032	1,663	32	31,451	$C_{sample}<C_{LCSW}$
OD76	4,026	200	13,058	724	$C_{sample}<C_{LCSW}$	3,637	8
TF74	18	55	$C_{sample}<C_{LCSW}$	16,821	19,696	$C_{sample}<C_{LCSW}$	15
n	11	13	9	11	12	9	8
Arith. mean	2,324	588	10,334	2,607	2,201	9,284	66
Min.	18	55	3,025	45	1	2,333	8
Max.	13,156	1,260	32,032	16,821	19,696	31,451	175

- **Estimated TOC (mg/L)**: The arithmetic mean TOC for Category III effluents is 3,336 mg/L, with a maximum of 18,026 mg/L for YO64 (**Figure 9; Table 12**). The lowest TOC estimate was observed for IS88 (154 mg/L).

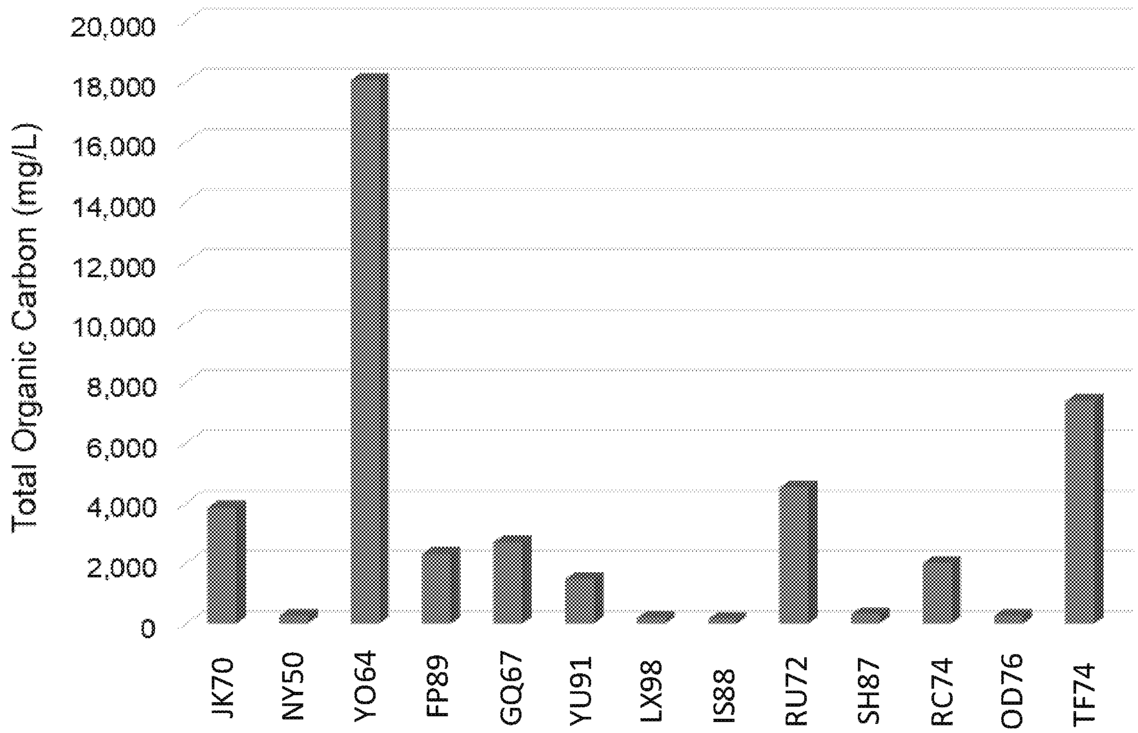

Figure 9. Bar chart of estimated TOC in undiluted TCW Category III effluents.

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Table 12. Raw Estimates and Descriptive Statistics for Estimated TSS and TOC in TCW Category III Effluents.		
Parameter	**TSS**	**TOC**
Units	mg/L	mg/L
JK70	4,872	3,846
NY50	4,205	256
YO64	19,641	18,026
FP89	4,564	2,308
GQ67	2,500	2,700
YU91	5,561	1,463
LX98	2,750	179
IS88	2,154	154
RU72	5,278	4,500
SH87	2,727	303
RC74	37,200	2,000
OD76	4,103	256
TF74	4,821	7,375
n	13	13
Arith. mean	7,721	3,336
Min.	2,154	154
Max.	37,200	18,026

- **Estimated TSS (mg/L)**: The highest estimated concentration of TSS was reported for RC74 (37,200 mg/L), and the lowest concentration was reported for IS88 (2,154 mg/L) (**Figure 10**).

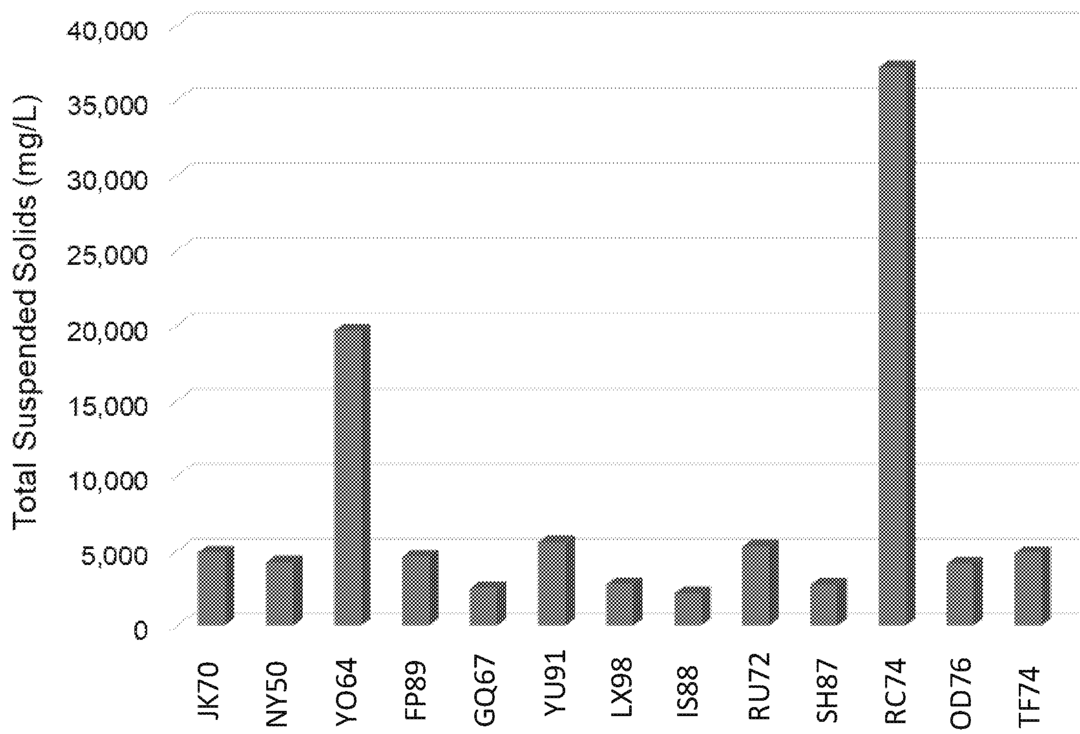

Figure 10. Bar chart of estimated TSS in undiluted TCW Category III effluents.

4.5 Variability in Effluent Composition During a Single Discharge

Variability in effluent chemical composition associated with well operation type and discharge duration was evaluated for select structures. Samples prepared at the CD and undiluted samples were used in the evaluation. The purpose of this evaluation is to show how effluent composition changes during TCW effluent discharges and addresses the question posed by USEPA Region 4 and Region 6 "*Does effluent composition change during the discharge?*"

4.5.1 Approach

Samples selected for the evaluation were collected from treatment and completion operations: EP57 and TR84 (completion); and RC74, OD76, and TF74 (treatment). Effluent parameters selected for evaluation were HCO_3^-, TOC, salinity, DOC, major cations (Ca^{2+}, Mg^{+2}, K^+, and Na^+) and major anions (Br^-, Cl^-, and SO_4^{2-}). The change in concentration of salinity, major cations and anions, TOC, and DOC over the discharge was expressed as a ratio (end:start). Non-detect values were represented by 100% of the laboratory RL when calculating the ratio. For the purpose of this discussion, effluent parameters that exhibit an increase with a ratio $\geq$2.0, or a decrease with a ratio $\leq$0.5 are emphasized. Ratios from 0.5 to 2.0 are assumed to reflect random variability.

4.5.2 Assessment Results

Assessment results indicate there is some variability in effluent composition when measured over the duration of a single discharge. Not all samples and parameters, however, were equally variable. Ratios for all parameters are presented in **Table 13**; results are summarized below:

- **EP57 and TR84:** The concentrations of the selected parameters were largely unchanged over the discharge. Sample EP57 was collected at the beginning of the discharge, and TR84 was collected at the end of the discharge, when the well stopped producing. This discharge structure had end-of pipe treatment, e.g., filtration and GAC.
- **RC74, OD76, and TF74**: This was a treatment operation. Effluents discharged included a Category III gel followed by a $CaCl_2$ brine with a small amount of ceramic proppant. Sample RC74 was collected at the beginning of the discharge, OD76 was collected in the middle, and sample TF74 was collected at the end of the reverse-out. Except for a decrease in salinity, differences in effluent composition between the beginning and the middle of the discharge were not pronounced. Substantial differences in effluent composition, however, were observed between the beginning and end of the discharge, and the middle and end of the discharge. The most noticeable changes were an increase in Br^-, total and dissolved Ca^{2+}, and salinity. The increases in Ca^{2+} and salinity likely reflect the shift to a $CaCl_2$ brine at the end of the discharge. It is not known what contributed to the large increase in Br^-.

Table 13. Change in Analytical Parameters at Different Times in the Discharge Expressed as a Ratio (End:Begin). Ratios ≥2.0 or ≤0.5 are boldfaced for clarity.

Parameters	Ratios			
	TR84 (End): EP57 (Begin)	OD76 (End): RC74 (Begin)	TF74 (End): RC74 (Begin)	TF74 (End): OD76 (Begin)
Water Quality Parameters				
HCO_3^- (Estimated as 1.22 * Total Alk.)	1.0	1.0	1.1	1.0
Organic Carbon, Total	1.0	1.0	1.0	1.0
Salinity (100% Effluent)	1.4	**0.3**	**6.3**	**18.1**
Dissolved Organic Carbon	1.0	1.0	1.1	1.1
Metals (Total)				
Ca	1.0	0.9	**8.0**	**8.9**
Mg	1.0	0.9	0.9	1.1
K	1.0	1.0	1.0	1.0
Na	1.0	0.9	1.0	1.0
Metals (Dissolved)				
Ca	1.0	1.1	**7.8**	**6.8**
Mg	1.0	1.1	0.9	0.8
K	1.0	1.0	1.0	0.9
Na	1.0	1.1	0.9	0.8
Inorganic Anions (Total)				
Br	1.4	0.9	**228.1**	**241.1**
Cl	1.0	1.0	1.0	1.0
$SO_4^{(2-)}$	0.9	1.1	1.0	0.9

4.6 Summary

Section 4.0 characterized TCW effluent composition for well treatment, completion, and workover operations. Based on the information provided, the JIP study questions identified at the beginning of Section 4.0 can be addressed as follows:

- *What are the concentrations of substances in GOM surface waters at the critical effluent dilution, i.e., the concentration predicted to exist in the effluent plume at the edge of the 100-meter (m) mixing zone?* The data evaluations conducted in support of this study question support the following observations:
    - Category I and III effluents at the CD are comprised of metals, cations and anions, and organics. Due to low critical dilution concentrations, ionic concentrations at the CD largely reflect the concentrations in laboratory control seawater.
    - Concentrations of some substances are highly variable, reflecting changes in TCW fluid composition needed to achieve operational objectives:
        - For substances in Category I effluents with a CV greater than 100%, the maximum concentration of Br^- was 2,630 mg/L. The maximum concentration of TOC was 406 mg/L, and 385 mg/L for DOC. The maximum concentration of Tl was 0.008 mg/L, and the maximum concentration of Cu was 0.046 mg/L.
        - For substances in Category III effluents with a CV greater than 100%, the maximum concentration of Br^- was 8,850 mg/L and 2,370 mg/L for total Ca^{2+}. The maximum concentration of dissolved As was 0.288 mg/L. The maximum concentration of COD was 580 mg/L, 70.3 mg/L for TOC, and 126 mg/L for DOC (126 mg/L).

- *What is the typical chemical composition of discharged TCW effluents?* The data evaluations conducted in support of this study question support the following observations:
  - Undiluted Category I effluents were denser than seawater due to elevated salinity and can be alkaline, with effluent pH reaching 10 S.U. Although also denser than seawater, Category III effluents were less saline than Category I effluents. Category III effluents exhibited higher TOC and TSS than Category I effluents.
  - Variability in effluent chemical composition was observed when evaluated over the duration of a discharge. The evaluations indicate that effluent composition is influenced by the type of well operation, in addition to the individual stages of a well operation. Additional factors that may have influenced the results include differences in the CD, use of chemical products, and brine type.

5.0 Acute Aquatic Toxicity of Discharged TCW Effluents

This section describes the acute whole effluent toxicity of Category I and Category III TCW effluents. The evaluations presented address the JIP study question "*How toxic are TCW effluents towards marine biota?*" Topics discussed are acute 48-h static renewal WET test procedures, preparation of Category III gel samples for WET testing, acute WET test results for TCW Category I effluents, WET test results for Category III effluents, and differences in the acute toxicity of Category I and III effluents. **Appendix D** presents results for a sample that was collected but that was later determined to have not been discharged and that was not representative of TCW effluents.

5.1 Acute 48-h Static Renewal WET Test

Acute, static renewal 48-hour WET testing was conducted consistent with the study plan, the GPs, and the USEPA (2002) guidance on WET methods. The WET test was used to evaluate the aggregate toxicity resulting from the mixture of all substances contained in the effluent.

5.1.1 WET Test Procedures

WET testing was conducted by EEUSA. WET test procedures are summarized below:

- **Test duration**: WET test organisms were exposed to the test medium for 48 hours (48-h).
- **Effluent dilution series**: Consistent with the study plan, the tested effluent dilutions were a laboratory control (0%); 0.1%; 0.3%; 0.8%; 2%; 6%; 18%; and 50%. The range of dilutions was chosen because historical WET testing of GOM produced water samples indicates (anecdotally) that complete mortality occurs at 100% effluent. The 0.1% effluent dilution reflects the anticipated lower limit of the critical effluent dilution. See Section 4.0 for a discussion on how the CD was calculated.
- **Test species and number of replicates**: The WET test species were *Americamysis bahia* (Mysid) and *Menidia beryllina* (Inland silverside minnow). A minimum of five (5) replicates with eight (8) organisms per replicate were used in the laboratory control and in each effluent dilution.
- **WET test endpoints:** Acute survival was evaluated. Test endpoints were a 48-hr no observed effect concentration (NOEC) and a lowest observed effect concentration (LOEC). Two supplemental test endpoints are a 25% lethal concentration (LC25) and a 50% lethal concentration (LC50).
- **WET test acceptability criteria (TAC)** are consistent with the GPs and USEPA (2002).
- **WET test holding time compliance:** WET test sample holding time was 36 hours from the time the TCW effluent sample is collected in the field, to the time of WET test setup at EEUSA. Sample holding times for three samples were exceeded due to transport delays (RU72), and the need to prepare difficult to analyze Category III gel samples (YO64 and YU91). Consistent with the study plan, samples exceeding the hold times were analyzed and reported, but the limitations of using such data were noted in the laboratory report. WET test holding time exceedances were also discussed with EEUSA.

- **Reference toxicant tests** were used by EEUSA to demonstrate the ability to obtain consistent results with the test method and evaluate the overall health and sensitivity of test organisms over time.

5.1.2 Preparation of Category III Gel Samples

Gel samples YO64, YU91, and OD76 required sample mixing because an aqueous solution is required to conduct WET testing. USEPA approved the adoption of the mixing approach as a departure from the original study plan via email on November 18, 2020. The gel samples were mixed by EEUSA with laboratory control seawater (LCSW) at 320 revolutions per minute (RPM) for 5 hours on magnetic stirrers using ½ inch diameter by 3-inch-long stir bars. Photographs of the mixing apparatus and an example of the aqueous solution after mixing the gel sample are presented in **Figure A3**.

5.1.3 Aquatic Toxicity of Category I Effluents

The aquatic toxicity of Category I effluents was variable. Variability in the observed toxicity reflects differences in well operation and in ZG57, EP57 and TR84, the presence of end-of-pipe treatment. Details are provided below by WET test organism:

- **Inland silverside minnow**: The arithmetic mean LC50 for the Inland silverside minnow was 12% effluent, with LC50s ranging from 0.6% to >50% effluent (**Table 14**). The most toxic TCW Category I effluent sample (LC50 = 0.6%) was AU71, which is a completion brine that contained chemical products. The least toxic sample (LC50 >50%) was ZG57; this long-term flowback effluent was treated with GAC and filtration.
- **Mysid**: The average LC50 for the Mysid was 9% effluent, with LC50s ranging from 0.54% to 35% effluent (**Table 14**). The most toxic sample (LC50=0.54%) was sample HV63, which is an effluent consisting of a $CaCl_2$ completion brine. No chemical products were present in HV63. The least toxic sample (LC50=35%) was sample ZG57.

Table 14. Acute 48-h whole effluent toxicity of TCW Category I Effluents.

Sample	WET Test Endpoint (% Effluent)							
	Inland silverside minnow				Mysid			
	NOEC	LC25	LOEC	LC50	NOEC	LC25	LOEC	LC50
HV63	2	3.05	6	4.11	0.3	0.42	0.8	0.54
RD67	2	3	6	4	0.3	0.46	0.8	0.61
RU61	0.8	1.51	2	2.54	0.3	0.44	0.8	0.57
XP62	2	2.92	6	3.95	0.3	0.44	0.8	0.57
LC54	2	3	6	4	2	2.94	6	4.12
AU71	0.3	0.45	0.8	0.6	0.3	0.46	0.8	0.66
ZG57	50	>50	>50	>50	18	26.5	>50	35.2
EP57	6	13.5	18	23.3	18	21.8	50	31.2
TR84	6	11	18	16	2	6	6	10

5.1.4 Aquatic Toxicity of Category III Effluents

Acute toxicity of Category III effluents was variable. TCW Category III gel samples were the most toxic TCW effluents sampled. TCW Category III sample IH80 could not be evaluated by the planned WET test protocols because it formed two phases in the laboratory upon mixing with LCSW. It was later determined that IH80 was not discharged so its properties are not characteristic of discharged TCW fluids. The toxicity of IH80 was evaluated with an alternative procedure (**Appendix D**). Details for TCW Category III effluents are provided below by WET test organism:

- **Inland silverside minnow**: The arithmetic mean LC50 for the Inland silverside minnow was 9.2% effluent, with LC50s ranging from 0.2% to 33.6% effluent (**Table 15**). The most toxic samples were YO64, which is a gel that contained several chemical products, and sample TF74. Sample TF74 was collected at the end of a well treatment job and contained the highest calcium and bromide concentrations observed in Year 1, along with elevated COD. The least toxic sample was BT52, which is a Category III fluid that contained a linear gel with breakers and cross-linkers.
- **Mysid**: The arithmetic mean LC50 for the Mysid was 3.5% effluent, with LC50s ranging from 0.05% to 13.1% effluent (**Table 15**). The most toxic sample was YO64, and the least toxic sample is SH87. Effluent sample SH87 was a well treatment operation in which chemical products were used.

Table 15. Acute 48-h whole effluent toxicity of TCW Category III Effluents. Notes: [1]. Fracturing gel. A homogeneous mixture suitable for WET testing was achieved after mixing (see Section 5.1.2 and Figure A3). [2] Sample contained proppant beads. The beads were removed before WET testing.

Sample	Difficult to Analyze Sample?	WET Test Endpoint (% Effluent)							
		Inland silverside minnow				Mysid			
		NOEC	LC25	LOEC	LC50	NOEC	LC25	LOEC	LC50
JK70	No	0.8	2.3	2.6	3.57	0.8	1.24	2.6	1.69
NY50	No	6	9	18	12	2	3	6	4
YO64	Yes	0.1	0.14	0.3	0.2	<0.1	0.03	0.1	0.05
FP89	No	0.3	0.41	0.8	0.54	<0.1	0.06	0.1	0.13
GQ67	No	0.8	1.05	2	1.37	0.3	0.43	0.8	0.56
YU91	Yes.[1]	6	9.64	18	13.3	0.1	0.15	0.3	0.2
LX98	No	2	3	6	4	0.8	1.1	2	1.4
IS88	No	0.8	1.1	2	1.4	0.3	0.55	0.8	0.8
RU72	Yes.[2]	0.3	0.45	0.8	0.6	0.8	1.08	2	1.39
BT52	No	18	25.4	50	33.6	6	9.08	18	12.2
SH87	No	18	26	50	34	6	9.53	50	13.1
RC74	No	6	9	18	12	6	8.82	18	12.4
OD76	Yes.[1]	6	8.77	18	11.9	<0.1	0.07	0.1	0.15
TF74	No	0.1	0.15	0.3	0.2	0.1	0.14	0.3	0.2

5.1.5 Comparison of TCW Category I and III Effluents

Differences in Mysid and Inland silverside minnow 48-h LC50s for Category I and Category III effluents were compared with a non-parametric Wilcoxon rank sum test. Statistically significant differences in 48-h LC50s are reported where $p \leq 0.05$.

Statistically significant differences were not observed between TCW Category I and Category III effluents for either species. The Mysid, however, is more sensitive to Category III effluents than the Inland silverside minnow and the difference is statistically significant ($p=0.007$) (**Figure 11**). Although TCW Category III effluents appear to be more toxic than Category I effluents, no statistically significant difference in 48-h LC50s was observed.

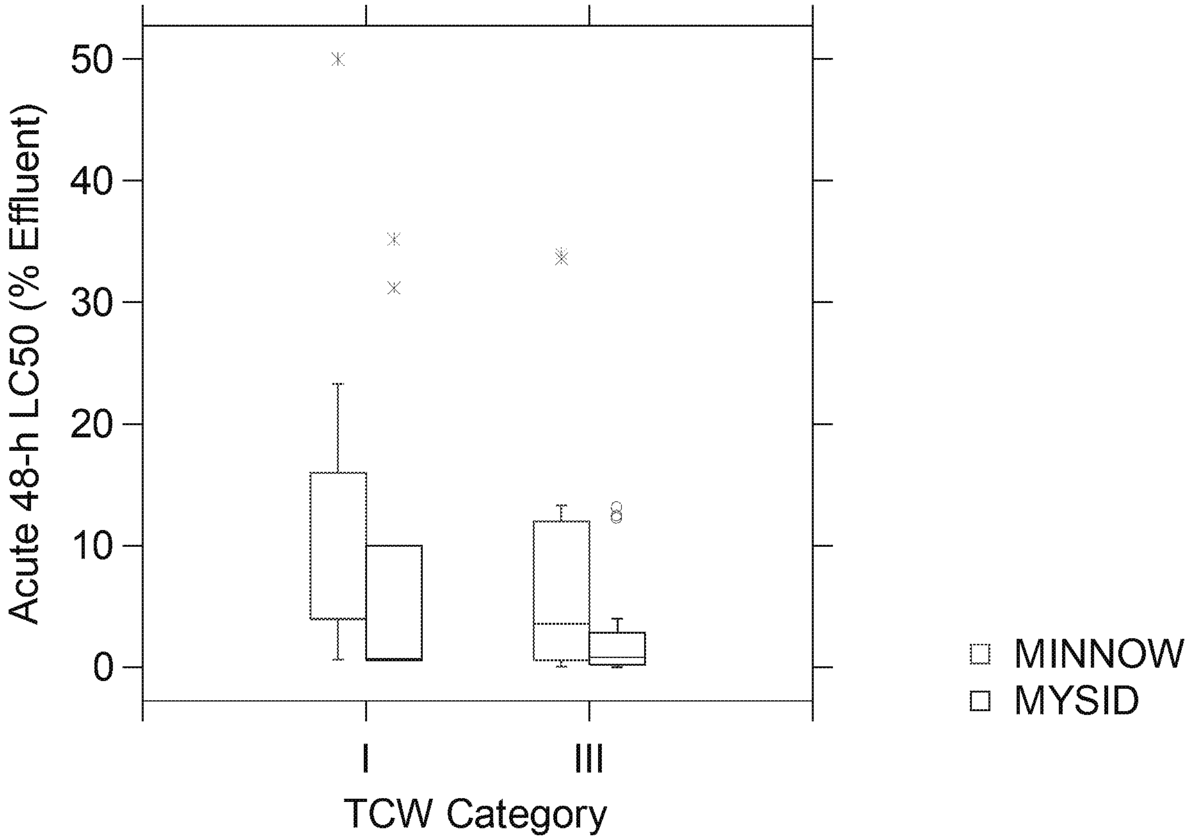

Figure 11. Boxplots for Mysid and Inland Silverside Minnow 48-H LC50s for TCW Category I (n=9) and TCW Category III effluents (n=14). The center line marks the median. Box edges are at the first and third quartiles. Whiskers show the range of observed values that fall within 1.5x of the interquartile range. Outliers (*), and extreme outliers (°) are shown. Additional details on boxplots are provided in **Appendix C**.

5.2 Summary

The evaluations presented in this section describe the acute 48-h whole effluent toxicity of Category I and Category III TCW effluents. The evaluations can be summarized as follows:

- TCW effluents exhibited a wide range of toxicities. The arithmetic mean LC50 for the Inland silverside minnow was 12% effluent, with LC50s ranging from 0.6% to >50% effluent. The arithmetic mean LC50 for the invertebrate Mysid was 9% effluent, with LC50s ranging from 0.54% to 35% effluent. This variability appears to be influenced by well operation, brine type, and the type(s) of chemical products used.
- Mysids were more sensitive to TCW Category III effluents than were the Inland silverside minnow, and the difference was statistically significant.

6.0 Acute Aquatic Hazard of Added Chemical Products

This section describes the acute aquatic hazard of added chemical products as proposed in the study plan. The evaluations presented address the JIP study question "*What are the general aquatic hazard characteristics of the substances currently used in TCW fluids?*"

Participants reported a total of 66 chemical products were used in formulating TCW fluids that were discharged to GOM surface water in Year 1. These products were typically mixtures and contained inorganic and organic substances that could potentially contribute to the observed acute whole effluent toxicity along with substances picked up as a result of circulation downhole. Examples of chemical classes include aldehydes, aliphatic amines, amides, cellulose ethers, phosphate esters, inorganic salts, neutral acids, neutral organics, and thiols/mercaptans.

The use of chemical products in the GOM by the oil and gas industry has been studied extensively. For example, the 2001 *Deepwater Program: Literature Review, Environmental Risk of Chemicals used in Gulf of Mexico Deepwater Oil and Gas Operations* study (Boehm et al., 2001) assessed risk to the aquatic environment associated with releases of chemical products. The study included an inventory of chemical products, and a summary of hazardous chemicals defined in 40 CFR 116 (Boehm et al., 2001). Of the 21 chemical products evaluated by Boehm et al. (2001) that could be identified from the SDSs, 5 were also used in the JIP study.

6.1 Hazard Assessment Approach

A simplified approach was used to qualitatively describe the aquatic hazard of chemical products. The manufacturer Safety Data Sheets (SDSs) provided by JIP study participants were used as the source of information for aquatic hazard. Concentrations of organic and inorganic substances in chemical products were not measured in the laboratory. This information is considered proprietary.

6.1.1 GHS Acute Aquatic Toxicity Classification

Safety Data Sheets for a minority of chemical products used in TCW fluids provided aquatic hazard information. For these products, an aquatic hazard assessment was conducted consistent with the United Nations (2019) guidance *A Guide to The Globally Harmonized System of Classification and Labeling of Chemicals (GHS) 8th Edition*. The GHS classification system provides an internationally recognized framework for assessing the level (or category) of aquatic toxicity hazard posed by a chemical product.

The acute GHS aquatic toxicity classification for a chemical product mixture was identified from SDS "*Section 2. Hazards Identification*". The provision of GHS aquatic toxicity data in SDS Section 2 is voluntary in the United States. For chemical products where GHS classification information was not provided in SDS Section 2, no aquatic hazard assessment could be made, and no conclusion about potential for aquatic toxicity is implied. These products were identified as "Not Assessed".

The GHS classification system for acute aquatic toxicity was applied to chemical products as follows (United Nations, 2019):

- **GHS Acute Category 1**: L(E)C50 $\leq$1.0 mg/L. Product is very toxic to aquatic life.
- **GHS Acute Category 2**: L(E)C50 >1.0 mg/L but $\leq$10 mg/L. Product is toxic to aquatic life.

- **GHS Acute Category 3**: L(E)C50 >10 mg/L but ≤100 mg/L. Product is harmful to aquatic life.

Where available, the Chemical Abstracts Service (CAS) No. and descriptive information on product composition presented in SDS *Section 3. Composition and Information on Ingredients* is used as a complement to the GHS acute aquatic toxicity category. Composition of individual substances is presented in % w/w.

6.2 TCW Category I Effluents

A total of 15 chemical products were identified as potentially being present in TCW Category I effluents. Chemical products are present in all but two TCW Category I effluent samples (**Table A6**). Eleven products used in TCW Category I effluents were identified as "Not Assessed". Four chemical products were assigned an acute GHS aquatic toxicity classification of 1-3 based on the description provided in SDS Section 2. The products provide chemical functionalities as biocides, defoamers, non-emulsifiers, and surfactants:

- "Biocide 1";
- "Defoamer 1";
- "Non-emulsifier 1"; and
- "Biocide 4".

6.2.1 GHS Acute Category 1

The single product with a GHS Acute Category 1 classification is "Biocide 1" and was present in sample LC54. This product was used as an electrophilic biocide and is comprised of glutaraldehyde (CAS No.111-30-8; 10-30% w/w) and methanol (CAS No. 67-56-1; 0.1-1 % w/w).

6.2.2 GHS Acute Category 2

Chemical products with a GHS Acute Category 2 classification are a defoamer and a non-emulsifier:

- **"Defoamer 1"**: The product was present in sample RD67 and is used to prevent or eliminate existing foam in water-based drilling fluids and brines. The product contains 30-60% w/w of an alkyl phosphate (tributyl phosphate or "TBP"; CAS No. 126-73-8). .
- **"Non-emulsifier 1"**: This product was present in samples RD67, RU61, LC54, and AU71. "Non-emulsifier 1" is used to prevent the formation of emulsions between calcium-based completion brines ($CaBr_2$ and $CaCl_2$) and crude oil. The product contains 30-60% w/w isopropanol (CAS No. 67-63-0), 5-10% w/w of ethylene glycol monobutyl ether (CAS No. 111-76-2), 5-10% w/w of a proprietary ammonium salt (CAS No. not provided), 1-5% w/w of proprietary quaternary ammonium compounds (QACs) (CAS No. not provided), 1-5% w/w of xylene (CAS No. 1330-20-7), and 0.1-1% w/w of methanol (CAS No. 67-56-1).

6.2.3 GHS Acute Category 3

The single products with a GHS Acute Category 3 classification is the lytic biocide "Biocide 4". This chemical product was present in sample AU71 and is used as a lytic biocide. “Biocide 4” is a cationic surfactant that contains 50% w/w of the QAC didecyldimethylammonium chloride (DDAC) CAS No. 7173-51-5, and two alcohols (ethyl [0-10% w/w; CAS No. 64-17-5] and methyl [30-40% w/w; CAS No. 67-56-1]).

6.3 TCW Category III Effluents

A total of 56 chemical products are potentially present in Category III effluent samples discharged to GOM surface water. These chemical products are present in all TCW Category III effluent samples (**Table A7**). Most products present in Category III effluents were identified as “Not Assessed” (n=47). A single product (“Oil Tracer 1”) had a chronic aquatic toxicity classification only.

There are more chemical products with the potential to contribute to aquatic toxicity than observed for Category I effluents. A total of 8 chemical products were observed in Year 1 with an acute aquatic toxicity GHS classification of 1-3. These chemical products provide chemical functionalities as biocides, corrosion inhibitors, non-emulsifiers, breakers, and oxygen scavengers:

- “Biocide 2”;
- “Biocide 3”;
- “Biocide 4”;
- “Corrosion inhibitor 1”;
- “Corrosion Inhibitor 5”;
- “Non-emulsifier 1”;
- “Breaker 1”; and
- “Oxygen Scavenger 1”.

6.3.1 GHS Acute Category 1

Two biocides and a corrosion inhibitor have a GHS Acute Category 1 classification:

- **“Biocide 2”**: This product was present in sample SH87 and is used as a water-based, non-oxidizing biocide in hydraulic fracturing treatment operations to minimize bacterial contamination. The product contains 1-5% w/w of the quaternary phosphonium biocide tributyl tetradecyl phosphonium chloride (TTPC) CAS No. 81741-28-8. This was the only substance identified in the SDS.
- **“Biocide 3”**: This product was present in sample FP89 as an electrophilic biocide to control bacterial growth. “Biocide 3” contains 60-100% w/w of the quaternary phosphonium compound tetrakis (hydroxymethyl) phosphonium sulphate (THPS) (2:1) (CAS No. 55566-30-8).
- **“Corrosion Inhibitor 5”**: This product was potentially present in sample SH87 and is used as an inorganic corrosion inhibitor intensifier. It contains 90-100% w/w copper acetate (CAS No. 6046-93-1).

6.3.2 GHS Acute Category 2

Chemical products with a GHS Acute Category 2 classification are a non-emulsifier, a corrosion inhibitor, and an emulsion breaker:

- **"Non-emulsifier 1"**: This non-emulsifier was present in samples NY50 and YO64. See discussion provided above for this chemical product.
- **"Corrosion inhibitor 1"**: This product was present in sample SH87 and was used as an acid corrosion inhibitor. Substances identified on the SDS are formic acid (40-50% w/w; CAS No. 64-18-6), an aromatic aldehyde (10-20% w/w; no CAS No. provided), oxyalkylated fatty acid (10-20% w/w; no CAS No. provided), an aromatic aldehyde (10-20% w/w; no CAS No. provided), a quaternary ammonium compound (QAC) (10-20% w/w; no CAS No. provided), isopropanol (5-10% w/w; CAS No. 67-63-0), a proprietary QAC (1-5% w/w; no CAS No. provided), methanol (1-5% w/w; CAS No. 67-56-1), 2-mercaptoethanol (1-5% w/w; CAS No. 60-24-2), and cyclic alkanes (1-5% w/w; no CAS No. provided).
- **"Breaker 1":** The product was potentially present in samples RC74, OD76, and TF74. This product is used as an emulsion breaker and contains sodium chloride (10-30% w/w; CAS No. 7647-14-5) and chlorous acid, sodium salt (5-10% w/w; CAS No. 7758-19-2). These are the only substances identified on the SDS.

6.3.3 GHS Acute Category 3

Chemical products with a GHS Acute Category 3 classification are a lytic biocide and an oxygen scavenger:

- **"Biocide 4"**: This product was potentially present in samples LX98 and IS88. See discussion provided above for this chemical product.
- **"Oxygen Scavenger 1"**: This product was used as a liquid oxygen scavenger for corrosion control of water-based fluids in TCW effluent samples LX98 and IS88. The product contains ammonium bisulfite (30-60%; CAS No. 10192-30-0); this is the only substance identified on the SDS.

6.4 Summary

The chemical hazard assessment qualitatively described acute aquatic hazard characteristics for chemical products. Performing more comprehensive evaluations would require proprietary information on the concentrations of individual substances in chemical products. The study question of "*What are the general aquatic hazard characteristics of the substances currently used in TCW fluids?*" can be addressed as follows:

- A total of 66 chemical products were potentially present in the TCW effluents sampled in Year 1. Approximately 83% of these chemical products were identified as "Not Assessed". For chemical products where GHS classification information was not provided in SDS Section 2, no aquatic hazard assessment could be made, and no conclusion about potential for aquatic toxicity is implied.
- Among the chemical products whose SDS presented GHS classifications, there were products in each of the three GHS acute aquatic toxicity categories: GHS Category 1 – Very toxic; GHS Category 2 – Toxic; and GHS Category 3 – Harmful.
- TCW Category III effluents contained more added chemical products than did TCW Category I effluents, including those with a GHS acute aquatic toxicity category of 1-3. The chemical functionalities of these products are electrophilic

and lytic biocides, cationic and non-ionic surfactants, breakers, corrosion inhibitors, non-emulsifiers, and defoamers.

- TCW chemical products contain primarily organic substances that could potentially contribute to aquatic toxicity in the TCW effluent samples. Substances of interest include the quaternary ammonium compounds (QACs), tributyl phosphate (TBP), and tributyl tetradecyl phosphonium chloride (TTPC). Products that contain these substances are used as cationic surfactants, lytic biocides, and non-emulsifiers.

7.0 Potential Causes of Acute Aquatic Toxicity

The evaluations presented in this section are used to address the study questions of "*Can general toxicity-composition connections be made?*" and "*What substances could potentially be associated with acute aquatic toxicity at the CD*?" Multiple lines of evidence were considered when assessing potential causes of acute aquatic toxicity. Evaluations conducted to assess the potential causes of acute aquatic toxicity are a statistical assessment of patterns in acute aquatic toxicity, toxicity-composition connection evaluations, and an acute aquatic toxicity screening at the critical effluent dilution.

7.1 Patterns in Acute Aquatic Toxicity

Patterns in acute toxicity were characterized by applying multivariate ordination to the WET test endpoint data for the Mysid and Inland silverside minnow. The purpose of the ordination was to assess potential differences and similarities in the acute toxicity of Category I and Category III effluents that could be used to support the toxicity-composition evaluations for both species.

7.1.1 Approach

Each Mysid and Inland silverside minnow WET test endpoint (NOEC, LOEC, LC25, and LC50), i.e., the "toxicity fingerprint" was ordinated with hierarchical and agglomerative cluster analysis. This approach addressed the relative sensitivity of the WET test species to substances in the TCW effluents. Details of the ordination and a separate ordination for the Mysid, which is the most sensitive WET test organism, are provided in **Appendix C**.

Cluster analysis is a multivariate procedure that was used to identify natural groupings in the individual WET test endpoint data. The cluster analysis yielded a dendrogram that grouped the TCW effluent samples according to similarity in WET test endpoints. The dendrogram was "cut" subjectively to yield meaningful clusters based on well operation, presence and absence of chemical products, and TCW effluent chemistry. For the purpose of the ordination, a default value of 0.035% effluent was assigned to WET test endpoints <0.1% effluent. WET test endpoints >50% effluent were defaulted to a value of 100% effluent.

7.1.2 Ordination Results

The dendrogram indicates that TCW Category I and Category III effluents did not ordinate into two separate groups, and that patterns in acute toxicity are driven by a set of factors more complex than effluent category (**Figure 13**). Nine clusters of effluent samples were identified (Clusters 1-9) that occur along an effluent toxicity gradient. Cluster 1 includes the most toxic samples, which are mostly Category III effluents. Cluster 9 contains the least toxic TCW Category I effluent. Samples were clustered based on the similarity of the Inland silverside minnow and Minnow WET test endpoints.

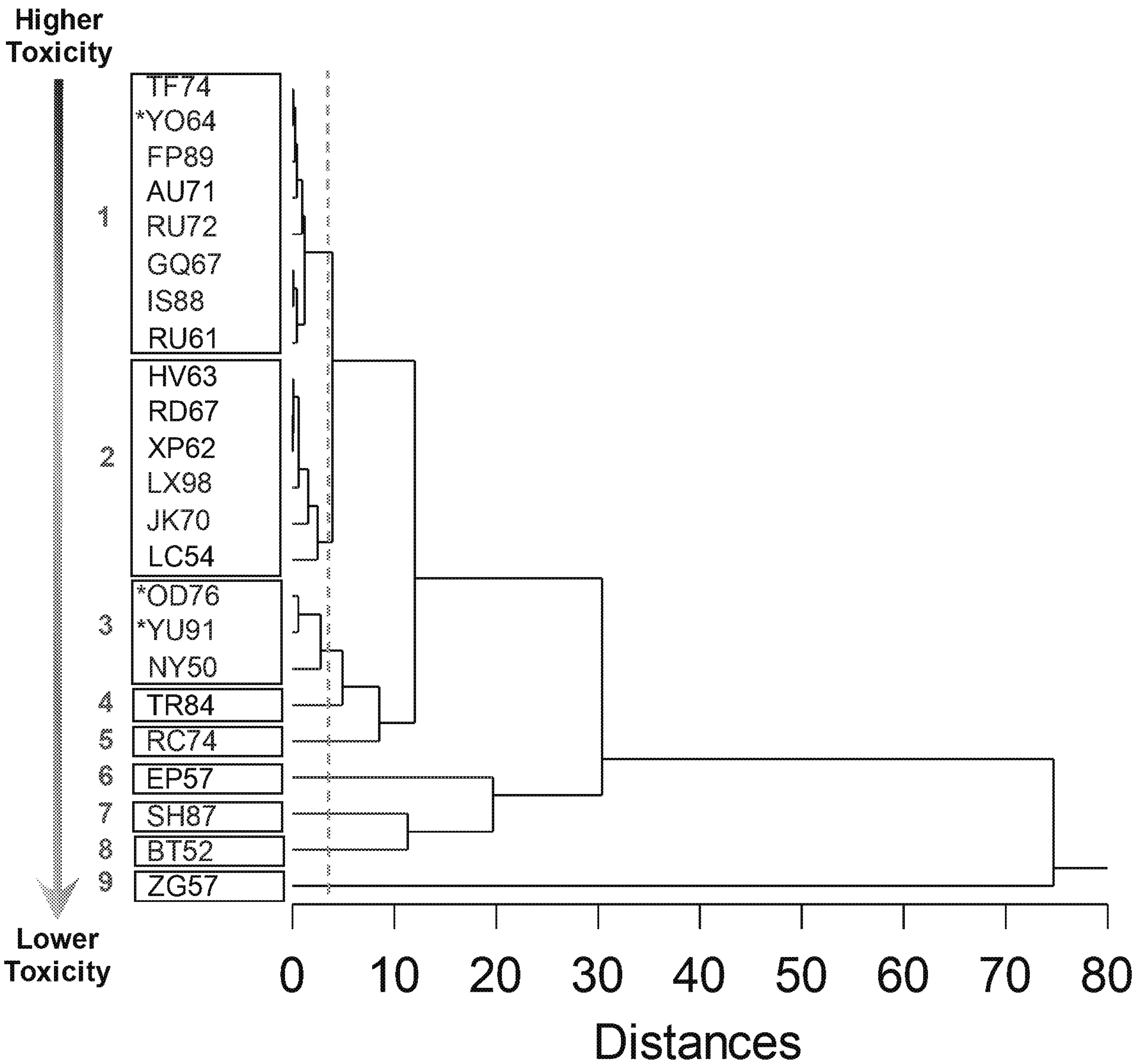

Figure 13. Cluster analysis dendrogram of the Inland silverside minnow and Mysid acute WET test endpoints (NOEC, LC25, LOEC, LC50). TCW Category I effluent samples are presented in black font, TCW Category III samples are in red font, and TCW Category III gel samples are denoted by a (*). The arrow illustrates a whole effluent toxicity gradient; and the vertical dashed blue line indicates where the dendrogram was "cut". Details of the ordination are provided in **Appendix C**.

Details of the dendrogram are provided below by cluster:

- **Cluster 1**: This cluster contains eight of the most toxic TCW effluent samples observed. Based on the LC50, the effluents are equally toxic to both WET test species: the arithmetic mean LC50 is 0.9% effluent for the Inland silverside minnow, and 0.5% for the Mysid (**Table 16**). The two most toxic samples are TF74 and YO64. Sample TF74 was collected from a treatment operation and contained the highest Br^- concentration observed. Based on the GHS classification, the sample also potentially contained chemical products comprised of substances that are potentially toxic to aquatic biota, e.g., GHS Acute

Category 2 emulsion breaker. Sample YO64 is a Category III gel sample that potentially contained several chemical products that are toxic to aquatic biota. These products are "Non-emulsifier 1" (GHS Acute Category 2) and the lytic biocide "Biocide 4" (GHS Acute Category 3), which both contain QACs. The TCW Category I effluent sample AU71 also contained these chemical products. Sample RU61 contained organic acids, "Non-emulsifier 1", and the highest concentrations of TOC and DOC observed during the study.

Table 16. Inland silverside minnow and Mysid Acute WET test endpoint by Cluster Analysis Grouping. An arithmetic mean is presented where n>1 and "n" represents the number of samples in the cluster.

Cluster	n	Inland silverside minnow (% Effluent)				Mysid (% Effluent)			
		NOEC	LC25	LOEC	LC50	NOEC	LC25	LOEC	LC50
1	8	0.4	0.7	1.1	0.9	0.3	0.4	0.7	0.5
2	6	1.8	2.9	5.4	3.9	0.8	1.1	2.2	1.5
3	3	6	9.1	18	12.4	0.7	1.1	2.1	1.5
4	1	6	11	18	16	2	6	6	10
5	1	6	9	18	12	6	8.8	18	12.4
6	1	6	13.5	18	23.3	18	21.8	50	31.2
7	1	18	26	50	34	6	9.5	50	13.1
8	1	18	25.4	50	33.6	6	9.1	18	12.2
9	1	50	100	100	100	18	26.5	100	35.2

- **Cluster 2:** Most of the samples in this cluster are TCW Category I effluents, with two TCW Category III effluents. The Mysid (arithmetic mean LC50 = 1.5% effluent) is approximately 3 times more sensitive to substances in the effluents than the Inland silverside minnow (arithmetic mean LC50 = 3.9% effluent). The most toxic samples in this cluster are TCW Category I effluent samples HV63 (completion operation) and RD67 (workover operation). Sample HV63 was a $CaCl_2$ brine that did not contain any chemical products, whereas RD67 contained "Defoamer 1" and "Non-emulsifier 1", both of which have a GHS Acute Category 2 classification.
- **Cluster 3:** This cluster consists of three TCW Category III effluents, two of which are gel samples. The Mysid (arithmetic mean LC50 = 1.5% effluent) is approximately 8 times more sensitive to substances in the effluents than the Inland silverside minnow (arithmetic mean LC50 = 12.4% effluent). Both NY50 and OD76 contain chemical products with a GHS Acute Category 2 classification.
- **Clusters 4 through 9:** The effluent samples in clusters 4 through 9 are the least toxic samples observed and include a mixture of TCW Category I and TCW Category III effluents. End of pipe treatment was present for the samples in clusters 4, 6, and 9; all samples were collected at the beginning of a long-term flowback. Samples in clusters 5, 7, and 8 were associated with treatment operations and exhibited low toxicity. Chemical products were present in these samples; it is possible that dilution with formation water may have occurred, contributing to the lower toxicity.

As discussed in **Appendix C**, the separate Mysid dendrogram also indicates that TCW Category I and Category III effluents did not ordinate into two separate groups. There is, however, a cluster of Category III effluents (including all gel samples) at the higher end of the toxicity gradient in the Mysid dendrogram. This suggests that Mysids are especially sensitive to the substances in TCW Category III effluents.

7.1.3 Summary

The ordination suggests that patterns in the acute aquatic toxicity of TCW effluents are complex and cannot be reduced to a single factor, e.g., TCW Category I effluent versus TCW Category III effluent. This complexity appears to be influenced by the type of brine and chemical products used. A summary of the data evaluations is provided below:

- TCW Category III effluents and a subset of TCW Category I effluents were the most toxic effluents sampled. This toxicity may be partially attributable to organic substances in chemical products, e.g., lytic biocides containing a cationic surfactant (didecyldimethylammonium chloride [DDAC]).
- The toxicity of samples in clusters containing a mixture of TCW Category I and TCW Category III samples is potentially influenced by both the cations or anions in brines, and organic substances.
- Except for Cluster 1, the Mysid is more sensitive to TCW effluents than the Inland silverside minnow.

The cluster analysis identified patterns in acute toxicity that may be explained by specific substances in the effluent. These substances are likely to be cations and anions from brines, and organics from chemical products.

7.2 Toxicity-Composition Connections

The toxicity-composition connections addressed the potential for aquatic hazard by addressing the following questions:

- *Do inorganic and organic substances potentially contribute to toxicity? Do Mysids and Inland silverside minnows respond differently to these substances?*
- *Are the observed toxicity-composition connections biologically plausible? Are they consistent with the current scientific literature?*

7.2.1 Approach

The data evaluations assess the contribution of inorganic and organic substances to the observed acute aquatic toxicity. The evaluations were conducted for TCW Category I and TCW Category III effluents. The approach consisted of selecting substances for evaluation, estimating concentrations of substances in 100% effluent, and data analysis. Details are provided below:

- **Acute toxic unit (TUa)**: The 48-h LC50s for Inland silverside minnow and Mysid were converted to an acute toxic unit (TUa) for the 23 effluent samples evaluated with 48-h WET testing. This approach normalized the LC50 to the whole effluent. The TU is defined by the USEPA (2010) as "*a measure of toxicity in an effluent as determined by the acute toxicity units (TUa) measured. The larger the TU, the greater the toxicity*". The USEPA (2010) calculates the TUa as "*100 times the reciprocal of the effluent concentration that causes 50 percent of the organisms to die in an acute toxicity test where TUa = 100/LC50*".
- **Substances selected for evaluation and rationale**: Substances were selected for evaluation based on their likely presence in effluents, potential toxicity towards the Mysid and Inland silverside minnow, and their ability to act as a

surrogate for organic toxicants. The substances selected are major cations and anions, TOC, and DOC. The rationale for their selection is presented below:

- **Dissolved cations/anions**: The cations evaluated are Ca^{2+}, K^{+}, Na^{+}, and magnesium (Mg^{2+}). The anions evaluated are HCO_3^-, Cl^-, and Br^-. Base brines of various densities are present in all effluent samples. As previously discussed, base brines used during the study are chloride brines: ($CaCl_2$, NaCl, and KCl), and bromide brines ($CaBr_2$ and NaBr).

  Toxicity towards marine organisms can result from an ion imbalance due to both deficiency and excess. Because TCW effluents are hypersaline, toxicity can be caused by an ion excess. Individual ions including K^{+}, Ca^{2+}, Mg^{+2}, and HCO_3^- have been shown to cause toxicity towards marine WET test organisms based on molarity models (Pillard et al., 2000). The Inland silverside minnow is more tolerant of ion-related toxicity than the Mysid (Pillard et al., 2000).

  Ion-related toxicity towards aquatic organisms can occur from exposure to individual ions, or an ion mixture. An ion imbalance can adversely affect osmoregulation. Although adverse effects may be associated with osmoregulation, individual ions can also adversely affect specific physiological function, which may be of greater significance (Pillard et al., 2000). For example, Ca^{2+} has been shown to be an important ion influencing Mysid toxicity (Kline and Stekoll, 2000), and in some cases was the primary cause of wastewater toxicity (Dorn and Rodgers, 1989; in Pillard et al., 2000).

- **TOC and DOC**: Organic substances may contribute to the observed toxicity. Because organic substances were not measured in sampled effluents, the water quality parameters of TOC and DOC were selected as a surrogate of organic substances in the effluent. Potential sources of DOC and TOC in TCW effluents include organic substances in chemical products, organic acids, residual hydrocarbons, and bacterial biomass.

- **Estimated concentrations of substances in 100% effluent (C_{TCW100}):** The laboratory analytical data measured at the CD were scaled to 100% effluent as previously discussed so that the analytical data could be related to the TUa. The need to infer the concentrations of substances in 100% effluent is a source of uncertainty.

- **Data analysis**: Correlation and regression analyses were conducted. Details are provided below:

  - Correlation: Due to issues of non-normality, non-parametric Spearman rank-order correlation was used to associate estimated concentrations of dissolved Ca^{2+} and total Mg^{+2}, Na^{+}, Br^-, Cl^-, and HCO_3^- in 100% effluent with the TUa. Statistically significant associations are reported where $p \leq 0.05$ consistent with Zar (1984) (see **Appendix C** for details).

  - Regression: Non-linear polynomial regression was used to characterize the association of the TUa with estimated concentrations of dissolved Ca^{2+}, DOC, and TOC in 100% effluent. A single data point was removed from the regression of Ca^{2+} and the TUa for TCW Category I effluents (AU71). Sample AU71 exhibited elevated Cl^- and low Ca^{2+}, yet high toxicity was observed for Inland silverside minnow and Mysid.

7.2.2 Acute Toxic Unit

The TUa is presented by sample in **Figure 12**. The sample with the highest Mysid TUa was the Category III gel sample YO64 (TUa = 2,000). The highest Inland silverside minnow TUa was 500 (YO64 and TF74). Relative to Category III effluents, the TUa is lowest for Category I TCW effluents with end-of-pipe treatment (TUa 2-10).

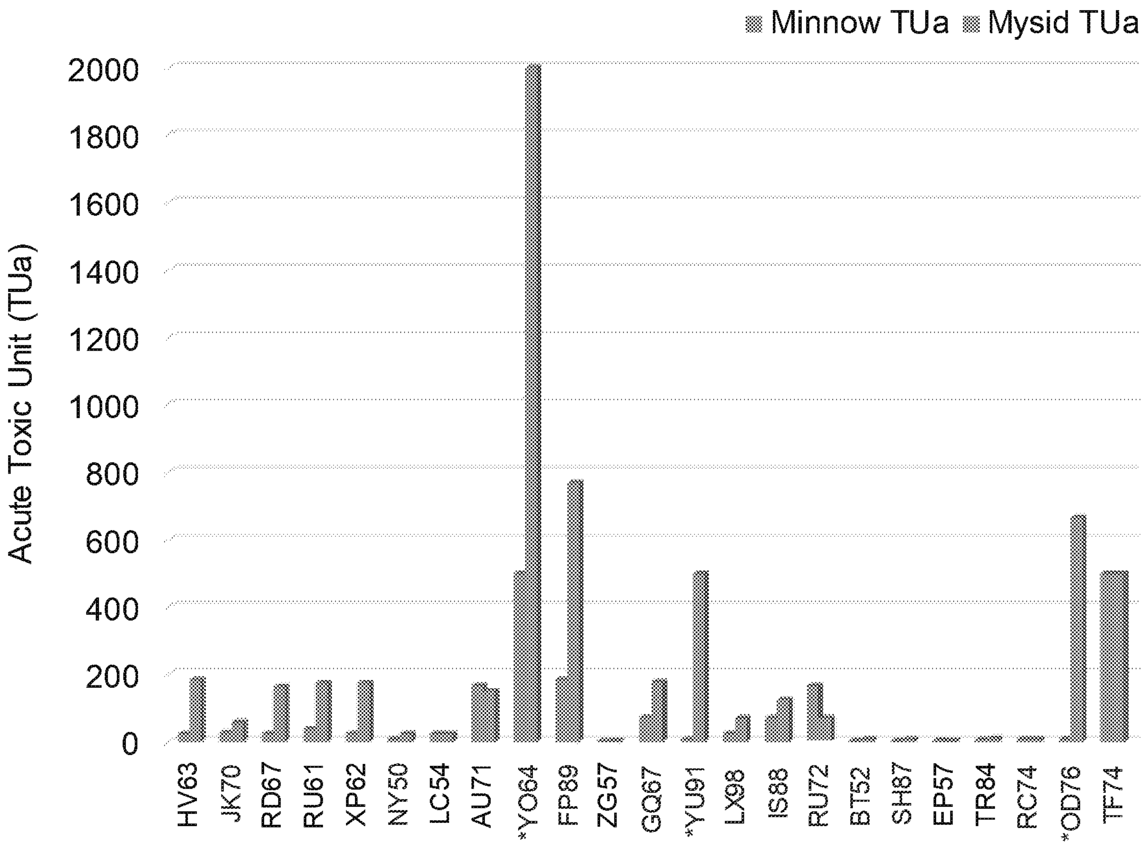

Figure 12. Acute toxic unit (TUa) by TCW effluent sample. The vertical bars represent the acute toxic unit where TUa = 100/LC50. A TUa of 2 indicates that the LC50 was 50% TCW effluent, whereas a TUa of 2,000 indicates that the LC50 was 0.05% TCW effluent. TCW Category I effluent samples are presented in black font, TCW Category III effluent samples are in red font, and TCW Category III gel samples are denoted by a (*).

7.2.3 TCW Category I Effluents

The cation Ca^{2+} is correlated with Mysid toxicity in TCW Category I effluents; the Inland silverside minnow is not as strongly influenced by Ca^{2+}. Of the ions evaluated, dissolved Ca^{2+} is the only substance that had a statistically significant positive association with the Mysid TUa (**Table 17**). That is, as Ca^{2+} increases, so does effluent toxicity. The identified association with Ca^{2+} is supported by the literature (Kline and Stekoll, 2000; Dorn and Rodgers, 1989; in Pillard et al., 2000). The correlation analysis indicates that there was no statistically significant association between the Inland silverside minnow TUa and any dissolved ion evaluated in TCW Category I effluents.

Table 17. Spearman rank-order Correlation of TUa and Estimated Dissolved Ion Concentration. TUa; acute toxic unit where TUa = 100/LC50. Boldfaced values indicate statistical significance ($p \leq 0.05$). Other values are not statistically significant ($p > 0.05$).			
Dissolved Ion (meq/L)	**Sample Size**	**Mysid TUa**	**Minnow TUa**
Ca	8	**0.79; p<0.05**	0.11
Mg	5	-0.6	-0.7
K	9	-0.46	-0.02
Na	8	0.01	0.07
Br	8	-0.24	-0.08
Cl	5	0.3	0.67
HCO_3^-	8	-0.43	-0.14

The regression also indicates that there is an association between dissolved Ca^{2+} (meq/L) and the Mysid TUa ($R^2 = 0.99$) (**Figure 14**). Dissolved Ca^{2+} (meq/L) was not as strongly related to Inland silverside minnow TUa ($R^2 = 0.59$) suggesting greater tolerance. The greater tolerance of the Inland silverside minnow to ion imbalance has been reported previously (Pillard et al., 2000).

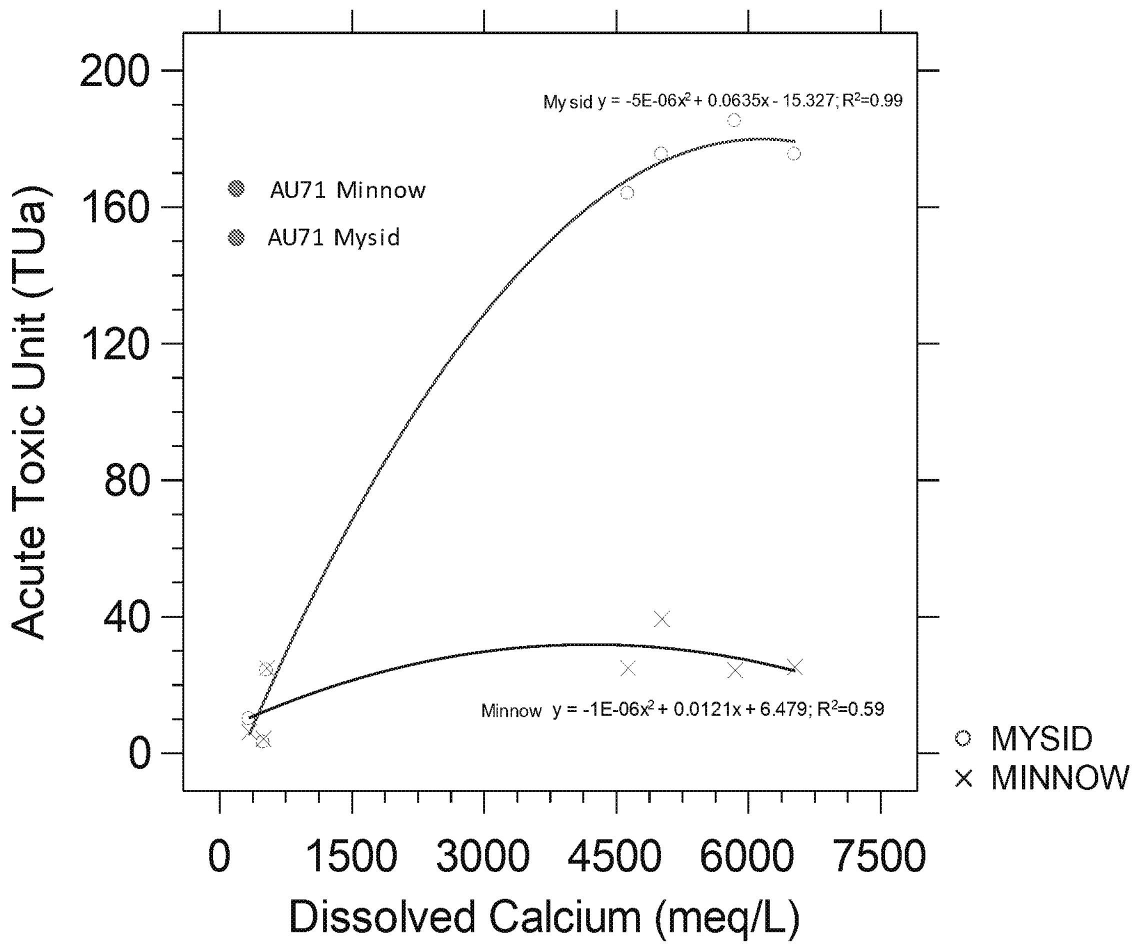

Figure 14. Polynomial regression of the Mysid and Inland silverside minnow TUa with dissolved calcium in TCW Category I effluents. Sample AU71 (●) was removed from the regression. AU71 exhibited elevated Cl^- and low Ca^{2+}, yet high toxicity was observed for Inland silverside minnow and Mysid. Sample ZG57 was also not included because $C_{sample} < C_{LCSW}$.

7.2.4 TCW Category III Effluents

There was no association between Ca^{2+} and the TUa for either the Inland silverside minnow or the Mysid exposed to TCW Category III effluents (**Figure 15**). This suggests that other substances were influencing toxicity in TCW Category III effluents.

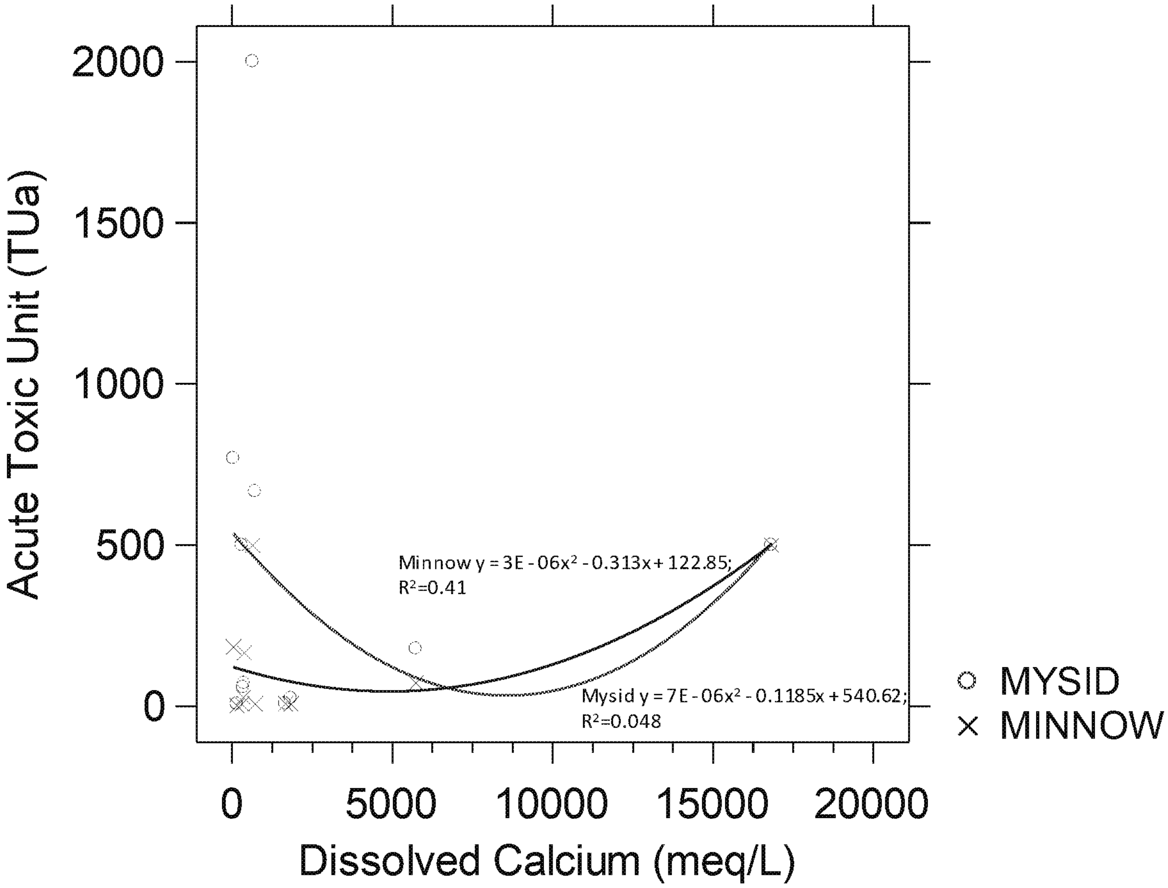

Figure 15. Polynomial regression of the Mysid and Inland silverside minnow TUa with dissolved calcium in Category III effluents.

A regression of Inland silverside minnow TUa suggests a stronger association with DOC and TOC than for Ca^{2+} (**Figure 16A**). There was more variability in the association of the Mysid TUa with DOC and TOC, however, and the regression is driven by a single data point (TUa = 2,000) (**Figure 16B**). This suggests that while organic substances may contribute to Mysid toxicity in TCW Category III effluents, which are complex, exposures likely involve more than one type of potential toxicant. This also raises the possibility that synergistic or antagonistic interactions might have occurred between toxicants with a different toxicological mode of action.

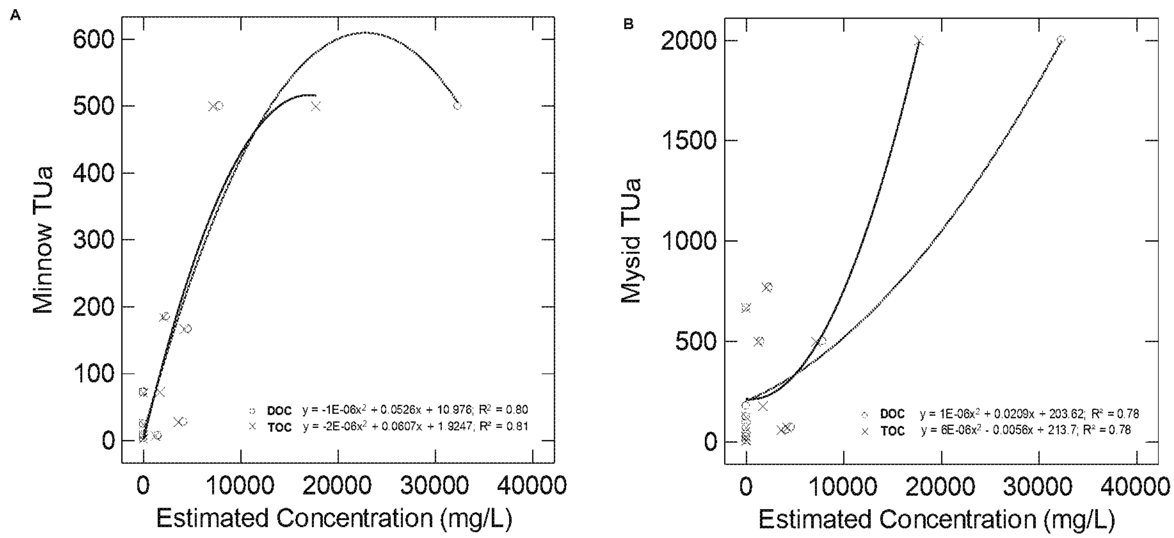

Figure 16. Polynomial regression of the Mysid and Inland silverside minnow acute toxic unit (TUa) with DOC and TOC in Category III effluents.

7.3 Acute Toxicity Screening at the CD

An acute toxicity screening was conducted to assess the potential risk of adverse effects towards aquatic biota at the CD and addressed substance concentrations predicted to exist at the edge of the 100-meter (m) TCW effluent mixing zone. The screening was conducted to address the study question "*What substances could potentially be associated with acute aquatic toxicity at the CD?*"

7.3.1 Acute Toxicity Screening Approach

The aquatic toxicity screening was conducted in two tiers. Tier 1 used conservative assumptions to identify key substances such as metals and major anions and cations in TCW effluents discharged to GOM surface waters. Such substances may have been used in formulating TCW fluids or may be picked up during downhole circulation. Tier 2 was included as a refinement step. As applied here, the purpose of Tier 2 is to focus the conclusions of the aquatic toxicity evaluations by identifying key substances. Details are provided below:

- **Tier 1**: The elements of the Tier 1 toxicity screening are provided below:
  - Exposure point concentration (EPC): Maximum concentrations of substances detected above the laboratory RL in any sample were identified. All 22 TCW effluent samples with laboratory analytical data were used. The arithmetic mean of the LCSW was subtracted from the TCW effluent maximum; 100% of the RL was used for non-detects identified in the LCSW. The resulting concentration was used as the EPC. If C_{sample}<C_{LCSW}, the substance was not considered further.
  - Ecological screening values (ESVs): ESVs are provided in **Table A8**. Substances with a USEPA published species-specific acute saltwater effects benchmark and/or aquatic life criterion were evaluated. Because the acute aquatic life criteria are intended to be protective of ≥95% of the aquatic community, published acute saltwater aquatic life criteria were

only used if reliable, species-specific effects benchmarks were not identified. The hierarchy of ESVs is as follows:

- Pillard et. al., 2000. Predicting the Toxicity of Major Ions in Seawater to Mysid Shrimp (*Mysidopsis bahia*), Sheepshead Minnow (*Cyprinodon variegatus*), and Inland Silverside Minnow (*Menidia beryllina*). The 48-h LC50s reported for Mysid and Inland silverside minnow were used.
- USEPA. 2018a. National Recommended Water Quality Criteria - Aquatic Life Criteria Table: Saltwater Criterion Maximum Concentration (CMC) (Acute).
- USEPA. 2018b. Region 4 Surface Water Screening Values for Hazardous Waste Sites: Saltwater (Acute).

- **Toxicity quotient (TQ)**: The TQ was used to qualitatively assess the association between the maximum concentration of a substance ($C_{CDMax.}$) and the potential for acute toxicity at the CD. The TQ was calculated where:

$$TQ = \frac{C_{CDMax.}}{ESV}; \text{ and}$$

  - TQ<1.0: If $C_{CDMax.}$is below the species-specific effects ESVs, then acute aquatic toxicity to Mysid and Inland silverside minnow is not probable. If there are no species-specific acute aquatic toxicity data, but $C_{CDMax.}$is below the aquatic life criterion, then it may be concluded that the constituent is likely not associated with acute toxicity to Mysid and Inland silverside minnow.
  - TQ≥1.0: If $C_{CDMax.}$is greater than or equal to the acute species-specific ESV (or aquatic life criterion if no species-specific ESV is available), this may indicate that the substance contributes to acute toxicity at the CD.

- **Tier 2**: Substances with an acute TQ≥1.0 were carried forward for Tier 2 refinement. An upper confidence limit (UCL) of the mean across all samples was used to refine the EPC. The USEPA software ProUCL (Ver. 5.1.002) was used to calculate the UCL (**Appendix E**). The arithmetic mean concentration in LCSW was subtracted from the UCL to generate the refined EPC. A negative value indicates that $C_{sample}<C_{LCSW}$.

7.3.2 Substances Potentially Contributing to Toxicity at the CD

Substances with TQs >1.0 are presented in **Table 18**. Specific substances include the anion Br^-, total, and dissolved metals: As, Ba, Ca^{2+}, Cu, Se, and Zn. Screening results are provided below:

- **Tier 1**: Samples containing substances with TQs >1.0 are nearly all Category III effluents, including two gel samples. Sample TF74 accounts for 40% of the 10 exceedances identified. TQs range from 1.0 (dissolved selenium) to 5.6 (total Cu). It is important to note that Cu was also detected above the ESV in the LCSW.

Table 18. Tier 1 Acute Toxicity Screening at the Critical Effluent Dilution: Exceedances. Category I effluent samples are presented in black font. Category III samples are in red font; Category III gel samples are denoted by a (*). The arithmetic mean LCSW was subtracted from the maximum to generate the EPC. "T" indicates total; "D" indicates dissolved.

Substance	Units	TCW Effluent Max.	Arith. Mean LCSW	EPC	TCW Sample with Max.	Frequency of Detection	ESV	Number of TCW Samples > ESV	Conc. in LCSW≥ ESV?	Toxicity Quotient (Max./ESV)
As, T	mg/L	0.181	0.055	0.126	SH87	2/22 (9%)	0.069	2/22	No	1.8
Ca, T	mg/L	2,370 2,370	267 267	2,103	TF74	22/22 (100%)	Mysid=1,100 Minnow=4,610	1/22	No	1.9 (Mysid)
Cu, T	mg/L	0.0550	0.0235	0.0315	TF74	13/22 (59%)	0.0056	13/22	**Yes**	5.6
Zn, T	mg/L	0.226	0.012	0.214	*YO64	7/22 (32%)	0.092	4/22	No	2.3
As, D	mg/L	0.288	0.055	0.233	RU72	2/22 (9%)	0.069	1/22	No	3.4
Ca, D	mg/L	2,140 2,140	258 258	1,883	TF74	22/22 (100%)	Mysid=1,100 Minnow=4,610	2/22	No	1.7 (Mysid)
Cu, D	mg/L	0.0460	0.0316	0.0145	EP57	5/22 (23%)	0.0048	5/22	**Yes**	3.0
Se, D	mg/L	0.47	0.17	0.29	RU72	17/22 (77%)	0.29	9/22	No	1.0
Zn, D	mg/L	0.36	0.06	0.30	*YO64	4/22 (18%)	0.09	2/22	No	3.3
Br,T	mg/L	8,850	38	8,812	TF74	22/22 (100%)	7,990	1/22	No	1.1 (Mysid)

- **Tier 2**: The Tier 2 refinements reduced the number of substances with TQs ≥1.0 to two metals that are essentially at the conservative ESV: dissolved As and total Cu (**Table 19**).

Table 19. Tier 2 Refinements of the Acute Toxicity Screening at the Critical Effluent Dilution. Notes: [1] The arithmetic mean concentration in LCSW was subtracted from the UCL to generate the refined EPC. A negative value indicates that $C_{sample}<C_{LCSW}$. [2] UCLs are computed across all TCW effluent samples. "T" indicates total; "D" indicates dissolved.

Parameter	Units	UCL[2] (mg/L)	UCL Type	Arithmetic Mean LCSW (mg/L)	Tier 2 EPC[1] (mg/L)	ESV (mg/L)	TQ
As, T	mg/L	0.025	KM H-UCL	0.055	-0.030	0.069	<1
Ca, T	mg/L	894	95% Chebyshev (Mean, Sd) UCL	267	627	1,100	<1
Cu, T	mg/L	0.0298	95% KM (t) UCL	0.0235	0.0063	0.0056	**1.1**
Zn, T	mg/L	0.099	95% KM (t) UCL	0.012	0.087	0.092	<1
As, D	mg/L	0.132	97.5% KM (Chebyshev) UCL	0.055	0.077	0.069	**1.1**
Ca, D	mg/L	835	95% Chebyshev (Mean, Sd) UCL	258	577	1,100	<1
Cu, D	mg/L	0.0232	Gamma Adjusted KM-UCL (use when k<=1 and 15 < n < 50 but k<=1)	0.0316	-0.0084	0.0048	<1
Se, D	mg/L	0.32	95% KM (t) UCL	0.17	0.15	0.29	<1
Zn, D	mg/L	0.10	95% KM (t) UCL	0.06	0.05	0.09	<1
Br,T	mg/L	2,386	95% Chebyshev (Mean, Sd) UCL	38	2,348	7,990	<1

7.4 Summary

The toxicity-composition connection evaluations presented in this section assessed whether patterns in acute toxicity are present, what some of the potential causes of toxicity are, and screened for substances that could potentially contribute to acute aquatic toxicity at the CD. The evaluations can be summarized as follows:

- Patterns in aquatic toxicity reflected the varying influence of organics and inorganics, i.e., mixture toxicity. This also raises the possibility that synergistic or antagonistic interactions might occur between toxicants with a different toxicological mode of action.

- TCW Category III gels were the most toxic effluents sampled. This toxicity is at least partially attributable to organic substances in chemical products.
- The least toxic samples are TCW Category I effluents with end-of-pipe treatment.
- The TCW effluents are complex and toxicity is likely occurring as a mixture. The cation Ca^{2+} appears to contribute to Mysid toxicity in TCW Category I effluents, whereas there is no association of Ca^{2+} with toxicity to the Inland silverside minnow. In TCW Category III effluents, DOC and TOC appear to contribute to Inland silverside minnow toxicity. Although organics are potentially influencing Mysid toxicity, the association with TOC and DOC is not as clear and other toxicants are likely playing a role.
- The Tier 1 aquatic toxicity screening identified exceedances for metals; the exceedances of the ESVs were primarily associated with TCW Category III effluents. The Tier 2 refined screening identified two metals, dissolved arsenic and total copper, whose UCL values were equal to the conservative ESVs. This suggests there is the potential for acute aquatic toxicity from these components at the edge of the mixing zone.

The identification of substances and discharge types that are potentially associated with the observed aquatic toxicity will support the Year 2 evaluations described in **Section 8.0**.

8.0 Recommendations for Year 2 JIP Study Activities

Recommendations for the Year 2 JIP study activities are presented in this section consistent with the study plan and reflect the Year 1 findings. The recommendation for Year 2 is to continue with the approach in Year 1. As discussed in the study plan, any refinements to the Year 2 JIP study activities will be discussed with USEPA Regions 4 and 6 before they are implemented.

8.1 Sample Collection Schedule

It is currently planned to collect TCW effluent samples from February to April 2021. This will accommodate the final report schedule, which is due to USEPA on October 1, 2021.

8.2 Sample Size

JIP Study participants were contacted in January 2021 to confirm the number and schedule of planned discharges. A streamlined survey questionnaire was submitted to the JIP study participants. The updated survey information will be used to select samples for 2021. LHS may be used to select samples in Year 2. Because they were effective at identifying a representative data set, the same LHS input parameters used in Year 1 will be carried forward for Year 2. At this time, the target sample size is 10, evenly divided between Category I and Category III effluents, if possible.

8.3 Laboratory Analysis

Consistent with the study plan, chemical analyses will be performed on samples diluted to the CD. All parameters measured in Year 1 will be analyzed. The sample mixing approach for WET testing of gel effluents, adopted as a USEPA-approved study plan change in Year 1, will continue to be used in Year 2. Additional LCSW samples will be analyzed for validation of concentrations in the synthetic laboratory control seawater.

8.4 Reporting

A final JIP study report presenting all Year 1 and Year 2 data will be prepared and submitted to USEPA on October 1, 2021, consistent with the study plan and the GP. The purpose of the report is to address the study questions regarding TCW discharge quality and the potential for TCW discharges to cause acute aquatic toxicity towards aquatic biota.

9.0 Conclusions

The Year 1 evaluations characterized TCW discharges and assessed the potential for TCW effluent characteristics to contribute to acute whole effluent toxicity. Year 1 efforts have led to a better understanding of TCW effluent characteristics, their aquatic toxicity, and substances that potentially contribute to this toxicity. The general Year 1 conclusions are as follows:

- **How toxic are TCW effluents?** TCW effluents exhibited a wide range of toxicities. The arithmetic mean LC50 for the Inland silverside minnow was 12% effluent, with LC50s ranging from 0.6% to >50% effluent. The arithmetic mean LC50 for the invertebrate Mysid was 9% effluent, with LC50s ranging from 0.54% to 35% effluent. A subset of TCW Category III effluents that formed gels were the most toxic effluents collected. The Mysid was generally more sensitive to TCW effluents than the Inland silverside minnow, and especially TCW Category III effluents.
- **What is contributing to the observed toxicity?** Multiple lines of evidence were used to identify individual substances and classes of substances potentially contributing to toxicity, and potential sources of these substances. Toxicity-composition evaluations of TCW effluents can be summarized as follows:
    - Ionic composition appears to be associated with the toxicity of TCW Category 1 effluents, specifically with Ca^{2+} concentrations, although this association is not definitive. Inland silverside minnow toxicity does not appear to be influenced by Ca^{2+} to the same extent as the Mysid.
    - Organics (based on the DOC and TOC concentrations used as surrogate for organic chemical products or organics picked up downhole) appear to contribute to Inland silverside minnow toxicity in most TCW Category III effluents. This association is not, however, definitive. Also, there does not appear to be as strong an association with Mysid survival, and other toxicants are likely influencing toxicity.
    - Ultimately, while inorganic and organic substances are likely contributing to the toxicity of TCW Category I and III effluents, which are complex, exposures likely involve more than one type of potential toxicant. This also raises the possibility that synergistic or antagonistic interactions might have occurred between toxicants with a different toxicological mode of action. The JIP study was not, however, designed to identify or account for these potential interactions.
- **Are there chemical products and/or products that are of interest?** A total of 66 chemical products were potentially present in the TCW effluents sampled in Year 1. These products can be summarized as follows:
    - Of the 66 chemical products reported as used in Year 1 samples, approximately 83% were identified as "Not Assessed". For chemical products where GHS classification information was not provided in SDS Section 2, no aquatic hazard assessment could be made, and no conclusion about potential for aquatic toxicity is implied.
    - Among the minority of chemical products whose SDS presented GHS classifications, there were products in each of the three GHS acute aquatic toxicity categories: GHC Category 1 – Very toxic; GHS Category 2 – Toxic; and GHS Category 3 – Harmful. For the majority of the

chemical products, no GHS data was presented in SDS and no assessment of hazard was conducted.

- TCW Category III effluents contained more added chemical products than did TCW Category I effluents, including those with a GHS acute aquatic toxicity category of 1-3. The chemical functionalities of these products are electrophilic and lytic biocides, cationic and non-ionic surfactants, breakers, corrosion inhibitors, non-emulsifiers, and defoamers.
- TCW chemical products contain primarily organic substances that could potentially contribute to aquatic toxicity in the TCW effluent samples. Substances of interest include the quaternary ammonium compounds (QACs), tributyl phosphate (TBP), and tributyl tetradecyl phosphonium chloride (TTPC). Products that contain these substances are used as cationic surfactants, lytic biocides, and non-emulsifiers.

The Year 2 sampling will occur from February to April 2021; the final report will be submitted to USEPA Region 4 and Region 6 in October 2021 consistent with the study plan. The recommendation for Year 2 is to continue with the approach in Year 1. Year 2 sampling will continue to use the sample mixing technique adopted as a USEPA-approved study plan change in Year 1 to conduct WET testing of gel effluents. Additional analyses of LCSW will be made to better define background concentrations. As discussed in the study plan, any refinements to the Year 2 JIP study activities will be discussed with USEPA Regions 4 and 6 before they are implemented.

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Appendix Tables

Table A1. TCW Effluent Sample Area, Block, and API Well No.

Sample ID	Area	Block	API Well No.
TCW-1	Mississippi Canyon	502AC	608174097300
TCW-2	Viosca Knoll	999	608174046000
TCW-3 (working with Operator to confirm API No.)	Walker Ridge	425	608234001600
TCW-4	Green Canyon	640	608114072600
TCW-5	South Timbalier	37	177154128600
TCW-6	Green Canyon	825	60811406960
TCW-7	Mississippi Canyon	809	608174112602
TCW-8A	Mississippi Canyon	807	608174047905
TCW-8B	Mississippi Canyon	807	608174047905
TCW-9	Mississippi Canyon	519	608174141100
TCW-10	Walker Ridge	718	60812401270
TCW-11	Mississippi Canyon	392	608174133401
TCW-12	Mississippi Canyon	520	608174139900
TCW-13A	Mississippi Canyon	807	608174048702
TCW-13B	Mississippi Canyon	807	608174048702
TCW-14	Ewing Bank	873	608105004901
TCW-15[1]	Mississippi Canyon	809	608174109102
TCW-16	Green Canyon	338	608114035403
TCW-17	Mississippi Canyon	807	608174048702
TCW-18A	Walker Ridge	758	608124012500
TCW-18B	Walker Ridge	758	608124012500
TCW-19A	Walker Ridge	508	608124012900
TCW-19B	Walker Ridge	508	608124012900
TCW-19C	Walker Ridge	508	608124012900

Notes:
[1] Sample TCW-15 was not discharged to surface water.

Table A2. TCW Effluent Discharge Characteristics

TCW Sample	HV63	JK70	RD67	RU61	XP62	NY50
Sample Collection Date	12/19/2019	11/8/2019	11/24/2019	1/2/2020	1/21/2020	2/8/2020
Job/Operation Type	Completion / Zonal isolation	Completion	Workover	Completion	Completion	Completion
TCW Fluid Category	I	III	I	I	I	III
TCW Fluid Description	CaCl2 Brine (10.5 ppg)	CaBr2 Brine (12.1 ppg)	CaCl2 Brine (11.6 ppg)	CaBr2 completion brine (12.1 ppg); KCl brine; HCl/Acetic acid treatment	CaCl2 Brine (10.5 ppg)	CaCl2 Brine (8.4 - 11.5 ppg); CaCl2-CaBr2 Brine 11-15 PPG
Time Discharge Commenced	1500	0650	2030	0015	0750	0540
Duration of Discharge (hours)	1.25	0.45	1.50	0.08	0.03	1.67
Pipe Diameter (inches)	18	18	12	4	6	8
Total Discharge Volume (bbl)	965	272	1,476	100	10	891
Discharge Rate (bbl/day)	18,528	14,400	23,616	28,800	7,200	12,830
Water Column Depth (ft.)	2,300[4]	4,119	8,832	4,250	62	4,976
Depth of Discharge (ft.) Relative to Water Surface	Not Reported	-15	-35	+50	+90	-12
Depth Difference (End-of-Pipe and Seafloor) (meters)	710[4]	1,251	2,681	1,311	46	1,513
CD (% Effluent)[5]	0.44	0.39	0.48	0.55	0.19	0.39
Is there Wastewater Treatment Before Discharge?	No	No	No	No	No	No.

Table A2. TCW Effluent Discharge Characteristics

TCW Sample	LC54	AU71	YO64[1]	FP89	ZG57	GQ67
Sample Collection Date	2/15/2020	3/14/2020	3/14/2020	3/18/2020	2/27/2020	4/24/2020
Job/Operation Type	Completion	Completion	Completion was open-hole with no fracturing	Treatment / Frac. job	Completion; Flow-back	Workover for plug/abandon. Cleaning Spacer/Soap Pill
TCW Fluid Category	I	I	III,gel	III	I	III
TCW Fluid Description	NaCl Brine (8.6 ppg)	NaCl brine; (9.5 ppg)	Gelled spacer between brines of differing weights.	Completion; Fracturing Fluid (SEAQUEST Linear Gel)	CaBr2 completion fluid w/cross-linker	Workover Spacer. 12.4ppg NaBr2 to 12.6 ppg completion CaBr2 brine.
Time Discharge Commenced	0600	1300	1300	0528	Ongoing discharge; sample collected at 0730	1940; 2030
Duration of Discharge (hours)	0.08	0.40	0.40	1.00	24; Although the total length of the flow back was 31 days, the JIP Study participant believed that the discharge of effluents occurred within the first 24 h.	1.67
Pipe Diameter (inches)	16	6.765	6.765	18	18	16
Total Discharge Volume (bbl)	320	189	189	473	2,534 bbls over the 31-day period; most of this volume was discharged in the first 2 days.	118
Discharge Rate (bbl/day)	92,160	11,340	11,340	11,352	A diffuser "duck bill" system is used. CORMIX modeling was conducted specifically for the platform. Discharge characteristics were not reported. The platform-specific critical effluent dilution of 0.291% was used.	1,699
Water Column Depth (ft.)	3,650	7,210	7,210	6,595		7,210
Depth of Discharge (ft.) Relative to Water Surface	-27	-12	-12	-36		-36
Depth Difference (End-of-Pipe and Seafloor) (meters)	1,104	2,194	2,194	1,999		2,208
CD (% Effluent)[5]	1.25	0.39	0.39	0.39	0.291	0.1
Is there Wastewater Treatment Before Discharge?	No	No	No	No	Yes. TCW fluids are sent through a treatment package of surge tanks; a weir box; solids filters; absorption media; and carbon vessels.	No.

Table A2. TCW Effluent Discharge Characteristics

TCW Sample	YU91[1]	LX98	IS88	RU72[2]	IH80[3]	BT52
Sample Collection Date	4/27/2020	4/20/2020	4/19/2020	4/30/2020	4/23/2020	5/1/2020
Job/Operation Type	Treatment; Frac. Job	Workover; Coiled tubing clean out-related fluid	Workover	Treatment	Treatment / Wellbore Cleaning spacer	Treatment
TCW Fluid Category	III, gel	III	III	III	III (not discharged to surface water)	III
TCW Fluid Description	A Completion/Cat III with 78% CaBr2 brine; SeaQuest lnear gel w/cross-linkers.Sample had a "Jell-O" like consistency.	Category III Workover - Packer Fluid – 8.5 ppg 2% KCl. According to Operator, the fluid has been present in the well for 19 years and was stored in a pit before discharge to surface water.	Packer fluid	Category III KCl brine frac-pack w/proppant. Linear gel. Proppant beads were identified in the sample container at a thickness of approx. 1-2 inches on bottom of container.	12 ppg CaBr2 (78% Sol.) Spacer chemicals in the sample include Baraklean 648 (17% solution) and Baraklean FL (4% solution). A separate phase was observed in the laboratory after settling for 24-h.	Category III frac. fluid brine / seawater; linear gel w/ breakers / cross-linkers.
Time Discharge Commenced	1105 (sample collected)	2204	0111	1315	Sample Not discharged to surface water. Discharge information not applicable.	2125
Duration of Discharge (hours)	1.50	0.40	0.18	0.42		1.08
Pipe Diameter (inches)	The discharge is through a 16" pipe that is flush with the underside of the ship's hull.	14	14	16		3
Total Discharge Volume (bbl)	498	543	543	118		256
Discharge Rate (bbl/day)	7,968	32,544	47,520	6,797		5,673
Water Column Depth (ft.)	6,700	2,955	2,955	773		3,325
Depth of Discharge (ft.) Relative to Water Surface	The current draft of the ship is the depth below the waterline at which the fluids are discharged (-36').	-15	-15	-15		+20
Depth Difference (End-of-Pipe and Seafloor) (meters)	2,031	896	896	231		1,020
CD (% Effluent)[5]	0.41	0.56	0.65	0.36		0.23
Is there Wastewater Treatment Before Discharge?	No.	No.	No	No.		No.

Table A2. TCW Effluent Discharge Characteristics

TCW Sample	SH87	EP57 - Begin	TR84 - Middle	RC74 - Begin	OD76 - Middle[1]	TF74 - End
Sample Collection Date	5/12/2020	5/10/2020	5/12/2020	5.24.20	5.25.20	5.25.20
Job/Operation Type	Treatment / Frac. job reversal	New well; Completion; Flow-back	New well; Completion; Flow-back	Treatment / Single Frac. Job; frac fluid reverse out	Treatment / Frac. Job; frac fluid reverse out	Treatment / Frac. Job; frac fluid reverse out
TCW Fluid Category	III	I	I	III	III, gel	III
TCW Fluid Description	Frac-fluid without radioactive tracers w/proppant	Operator indicated that TCW fluid use would be similar to TCW-10: CaBr2 and CaBr2 completion fluid w/cross-linker	Operator indicated that TCW fluid use would be similar to TCW-10: CaBr2 and CaBr2 completion fluid w/cross-linker	Frac. Gel with some Category III CaCl2 brine. No radioactive tracer.	Gel/Category III CaCl2 brine; Operator indicated that the sample may contain some proppant. No radioactive tracer.	The sample consists of a "cleaned-up" Category III CaCl2 brine with a small amount of proppant. No radioactive tracer.
Time Discharge Commenced	0957	1530	1530	2317 (sample collected at 2320); discharge ended at 0206	2317 (sample collected at 0124); discharge ended at 0206	2317 (sample collected at 0201); discharge ended at 0206
Duration of Discharge (hours)	3.38	1.42	16	0.05	2.07	0.60
Pipe Diameter (inches)	14	18	18	16	16	16
Total Discharge Volume (bbl)	568	132	2,087	30	1,211	1,577
Discharge Rate (bbl/day)	4,029	2,236	3,130	520	14,063	27,360
Water Column Depth (ft.)	2,940	A diffuser "duck bill" system is used. CORMIX modeling was conducted specifically for the platform. Discharge characteristics were not reported.	A diffuser "duck bill" system is used. CORMIX modeling was conducted specifically for the platform. Discharge characteristics were not reported.	9,558	9,558	9,558
Depth of Discharge (ft.) Relative to Water Surface	-12			-40	-40	-40
Depth Difference (End-of-Pipe and Seafloor) (meters)	892			2,901	2,901	2,901
CD (% Effluent)[5]	0.33	0.16	0.21	0.05	0.39	0.56
Is there Wastewater Treatment Before Discharge?	No.	Yes. A treatment package of surge tanks; a weir box; solids filters; absorption media; and granular activated carbon (GAC) vessels.	A treatment package of surge tanks; weir box; solids filters; absorption media; and GAC vessels. The GAC filters were "spent" when sample TCW-18B was collected.	No	No	No

Notes:
%; percent
CaBr; calcium bromide
NaCl; sodium chloride
ppg; pounds per gallon
TCW; treatment, completion, and workover
[1]. TCW Category III gel samples that require pre-mixing before conducting the standard acute WET test.
[2]. TCW Category III samples that require pre-preparation before WET testing including the removal of proppant beads.
[3]. TCW Category III samples that require an alternative toxicity test method to address the presence of a separate phase (Water Accommodated Fraction [WAF]). Sample was not discharged to surface water.
[4] Identified as 2,330' (implied feet) on the WET test sample chain of custody.
[5] CD; critical effluent dilution identified using the produced water tables identified in the USEPA Region 6 GP.

Table A3. Substances Potentially in Brines and Chemical Additives by TCW Effluent Sample

TCW Sample	TCW Cat. Type	Substances Potentially Present
HV63	I	$CaCl_2$ brine 10.5 ppg. Operator indicated that no chemical additives were used.
JK70	III	$CaBr_2$ brine; Misc. Amines/Quaternary Ammonium Salts; tributyl phosphate; isopropyl alcohol; glutaraldehyde; ethoxylated alcohol; ethylene glycol monobutyl ether; (2-(2-Methoxy methyl ethoxy)Methylethoxy) Propanol; Hydroxy ethyl cellulose; Xanthan Gum; Benzenesulfonic acid, C10-16-alkyl derivatives, compounds with 2-Propanamine; Dodecylbenzenesulfonic acid; 2-Ethylhexanol
RD67	I	$CaCl_2$ brine; $CaBr_2$ brine; tributyl phosphate; isopropanol; ammonium salt; quaternary ammonium compounds; ethylene glycol monobutyl ether; xylene; methanol
RU61	I	$CaBr_2$ brine 12.1 ppg; KCl brine; acetic acid; hydrochloric acid; isopropanol; ammonium salt; quaternary ammonium compounds; xylene; methanol; dipropylene glycol monomethyl ether
XP62	I	$CaCl_2$ brine 8.4 - 11.6 ppg
NY50	III	$CaCl_2$ brine, $CaBr_2$ brine; isopropanol; ethylene glycol monobutyl ether; ammonium salt; quaternary ammonium compounds; xylene; methanol
LC54	I	Glutaraldehyde; Methanol; isopropanol; ethylene glycol monobutyl ether; ammonium salt; quaternary ammonium compounds; xylene; methanol; NaCl
AU71	I	NaCl; ethylene glycol monobutyl ether; hydrotreated light petroleum distillate; D-Glucopyranose, oligomeric, decyl octyl glycosides; orange, sweet, extract; sodium hydroxide; isopropanol; ammonium salt; quaternary ammonium compounds; xylene; methanol; dipropylene glycol monomethyl ether; didecyldimethylammonium chloride (DDAC); ethyl alcohol; methyl alcohol
YO64	III,gel	Ethylene glycol monobutyl ether; hydrotreated light petroleum distillate; D-Glucopyranose, oligomeric, decyl octyl glycosides; orange, sweet, extract; sodium hydroxide; isopropanol; ammonium salt; quaternary ammonium compounds; xylene; methanol; dipropylene glycol monomethyl ether; DDAC; ethyl alcohol; methyl alcohol; NaCl
FP89	III	SeaQuest Linear Gel; tetrakis(hydroxymethyl)phosphonium sulphate(2:1); Hemicellulase enzyme; Cocamidopropyl betaine; Glycol ether; Guar gum; cationic polymer in solution; Ethoxylated alcohol; potassium carbonate; Hydrochloric acid; Acetic anhydride; Hydrofluoric acid; Xylene; Acetic acid; 2-Butoxyethanol
ZG57	I	$CaBr_2$ brine; kerosene; naphthalene; ethylbenzene; methanol; quaternary ammonium compound; fatty acid-amine condensate; ethylene glycol; 2-mercaptoethanol; oxyalkylate; diethanolamine; heavy aromatic naphtha; naphthalene; substituted alkylamine; 2-Butoxyethanol; sodium molybdate; inorganic salt; proprietary polyol compound; proprietary amine compound; proprietary diol compound 1&2; proprietary lactam compound;
GQ67	III	2.4 ppg $NaBr_2$ brine and 12.6ppg completion $CaBr_2$ brine; Tetraclean 107 (alcohols C9-11 ethoxylated, proprietary organic alcohol)
YU91	III, gel	Sodium carbonate; hydrochloric acid; acetic anhydride; hydrofluoric acid; acetic acid; NaCl brine 8.4-10 ppg; SeaQuest Linear Gel - Crosslinked ulexite; Water Frac H
LX98	III	Ammonium bisulfite; KCl brine
IS88	III	Ammonium bisulfite; sodium hydroxide; DDAC; ethyl alcohol; methyl alcohol
RU72	III	KCl brine, proppant beads present in sample (no SDS provided), Operator indicated a linear gel was present (no SDS provided).
BT52	III	Operator to provide SDSs.
SH87	III	Acetic acid; ammonium chloride; hydrogen chloride; hydrofluoric acid; tributyl tetradecyl phosphonium chloride; potassium carbonate; crystalline silica: cristobalite (proppant); formic acid; oxyalkylated fatty acid; aromatic aldehyde; quaternary ammonium compound; isopropanol; methanol; 2-Mercaptoethanol; cyclic alkanes; copper acetate; hemicellulase enzyme; citric acid; cocamidopropyl betaine (surfactant); sodium chlorite; ammonium persulphate; glycol ether; guar gum; alkoxylated alcohol; potassium iodide; NaCl; cationic polymer in solution (DNG); ethoxylated alcohol; xylene; acetic acid; 2-butoxyethanol; Poly(oxy-1,2-ethanediyl), a-undecyl-w-hydroxy-; glyoxal; sodium tetraborate; sodium hydroxide
EP57	I	See TCW-10; Operator indicated that the job type and chemical use is similar.
TR84	I	See TCW-10; Operator indicated that the job type and chemical use is similar.
RC74	III	$CaCl_2$ brine, Borate salts; dipropylene glycol monomethyl ether; diesel; ethylene glycol; methanol; ceramic materials and wares, chemicals (proppant); sodium hydroxide; T-803; soy methyl ester; oil tracer; chlorous acid; sodium salt; NaCl
OD76	III,gel	
TF74	III	

Table A4. Laboratory Analytical Parameters

Water Quality Parameters	Sample Type	Directly Measured or Estimated?
Total Dissolved Solids (Residue, Filterable)	Critical effluent dilution	Direct measurement
pH	Undiluted (100%) effluent	Direct measurement
Dissolved Organic Carbon (DOC)	Critical effluent dilution	Direct measurement
Alkalinity, Total (As CaCO3)	Critical effluent dilution / Undiluted (100%) Effluent	Direct measurement for both sample types
HCO_3^- (Estimated as 1.22 * Total Alk.)	Critical effluent dilution / Undiluted (100%) Effluent	Estimated
Nitrogen, Ammonia (As N)	Critical effluent dilution	Direct measurement
Hardness, Total (as CaCO3)	Critical effluent dilution	Direct measurement
Total Suspended Solids (Residue, Non-Filterable)	Critical effluent dilution / Undiluted (100%) Effluent	Direct measurement / Estimated
Chemical Oxygen Demand (COD)	Critical effluent dilution	Direct measurement
Sulfide	Critical effluent dilution	Direct measurement
Specific Gravity	Undiluted (100%) effluent	Direct measurement
Total Organic Carbon (TOC)	Critical effluent dilution / Undiluted (100%) Effluent	Direct measurement / Estimated
Metals (Total/Dissolved)		
As	Critical effluent dilution	Direct measurement
Ba	Critical effluent dilution	Direct measurement
Cd	Critical effluent dilution	Direct measurement
Cr	Critical effluent dilution	Direct measurement
Cu	Critical effluent dilution	Direct measurement
Pb	Critical effluent dilution	Direct measurement
Hg	Critical effluent dilution	Direct measurement
Ni	Critical effluent dilution	Direct measurement
Se	Critical effluent dilution	Direct measurement
Tl	Critical effluent dilution	Direct measurement
Zn	Critical effluent dilution	Direct measurement
Cations/Anions		
Br, Total	Critical effluent dilution / Undiluted (100%) Effluent	Direct measurement / Estimated
Ca, Total/dissolved	Critical effluent dilution / Undiluted (100%) Effluent	Direct measurement / Estimated
Cl, Total	Critical effluent dilution / Undiluted (100%) Effluent	Direct measurement / Estimated
Mg, Total/dissolved	Critical effluent dilution / Undiluted (100%) Effluent	Direct measurement / Estimated
K, Total/dissolved	Critical effluent dilution	Direct measurement
Na, Total/dissolved	Critical effluent dilution / Undiluted (100%) Effluent	Direct measurement / Estimated
SO_4^{2-}, Total	Critical effluent dilution	Direct measurement
Polycyclic Aromatic Hydrocarbons (PAHs)		
Acenaphthene	Critical effluent dilution	Direct measurement
Acenaphthylene	Critical effluent dilution	Direct measurement
Anthracene	Critical effluent dilution	Direct measurement
Benzo(a)anthracene	Critical effluent dilution	Direct measurement
Benzo(a)pyrene	Critical effluent dilution	Direct measurement
Benzo(b)fluoranthene	Critical effluent dilution	Direct measurement
Benzo(g,h,i)perylene	Critical effluent dilution	Direct measurement
Benzo(k)fluoranthene	Critical effluent dilution	Direct measurement
Chrysene	Critical effluent dilution	Direct measurement
Dibenzo(a,h)anthracene	Critical effluent dilution	Direct measurement
Fluoranthene	Critical effluent dilution	Direct measurement
Fluorene	Critical effluent dilution	Direct measurement
Indeno(1,2,3-cd)pyrene	Critical effluent dilution	Direct measurement
Naphthalene	Critical effluent dilution	Direct measurement
Phenanthrene	Critical effluent dilution	Direct measurement
Pyrene	Critical effluent dilution	Direct measurement

Table A5. Laboratory Analytical Results for Laboratory Control Seawater and TCW Effluent Samples

Constituent	Units	WET Lab Diluent-1	WET Lab Diluent-2
Critical Effluent Dilution	%	--	--
Date	--	11/11/2019	3/2/2020
Water Quality Parameters (Total)			
Hardness (as CaCO3)	mg/L	4,430	4,150
Alkalinity, Total (As CaCO3)	mg/L	55	92.5
HCO_3^- (Estimated as 1.22 * Total Alk.)	mg/L	67.1	112.9
Total Suspended Solids (Residue, Non-Filterable)	mg/L	ND<5.2	ND<5
Nitrogen, Ammonia (As N)[1]	mg/L	ND<0.5	ND<0.5
Chemical Oxygen Demand	mg/L	ND<300	ND<300
Organic Carbon, Total	mg/L	ND<2	ND<4
Sulfide	mg/L	ND<0.02	0.03
Specific Gravity	@4 ˚C	LCSW not analyzed.	LCSW not analyzed.
Water Quality Parameters (Dissolved)			
Total Dissolved Solids (Residue, Filterable)	mg/L	20,300	24,400
Dissolved Organic Carbon	mg/L	ND<2	ND<2
Metals (Total)			
As	mg/L	ND<0.01	ND<0.1
Ba	mg/L	0.022	ND<0.1
Cd	mg/L	0.002	0.013
Ca	mg/L	273	261
Cr	mg/L	ND<0.01	ND<0.1
Cu	mg/L	0.017	ND<0.03
Pb	mg/L	ND<0.005	ND<0.05
Mg	mg/L	910	848
Hg	mg/L	0.0000009	0.0000039
Ni	mg/L	ND<0.005	ND<0.05
K	mg/L	280	283
Se	mg/L	0.132	0.307
Na	mg/L	6,560	6,630
Tl	mg/L	ND<0.006	ND<0.06
Zn	mg/L	0.012	ND<0.1
Metals (Dissolved)			
As	mg/L	ND<0.01	ND<0.1
Ba	mg/L	0.0235	ND<0.1
Cd	mg/L	0.0022	ND<0.01
Ca	mg/L	256	259
Cr	mg/L	ND<0.01	ND<0.1
Cu	mg/L	0.0131	ND<0.05
Pb	mg/L	ND<0.005	ND<0.05
Mg	mg/L	837	848
Hg	mg/L	ND<0.0000005	0.0000011
Ni	mg/L	ND<0.005	ND<0.05
K	mg/L	243	278
Se	mg/L	0.147	ND<0.2
Na	mg/L	6,790	6,700
Tl	mg/L	0.0072	0.123
Zn	mg/L	ND<0.01	ND<0.1
Inorganic Anions (Total)			
Br	mg/L	37.5	37.6
Cl	mg/L	13,000	13,700
SO_4^{2-}	mg/L	1,830	2,070
Polycyclic Aromatic Hydrocarbons (PAHs)			
Acenaphthene	mg/L	ND<0.004	ND<0.004
Acenaphthylene	mg/L	ND<0.004	ND<0.004
Anthracene	mg/L	ND<0.004	ND<0.004
Benzo(a)anthracene	mg/L	ND<0.004	ND<0.004
Benzo(a)pyrene	mg/L	ND<0.004	ND<0.004
Benzo(b)fluoranthene	mg/L	ND<0.004	ND<0.004
Benzo(g,h,i)perylene	mg/L	ND<0.004	ND<0.004
Benzo(k)fluoranthene	mg/L	ND<0.004	ND<0.004
Chrysene	mg/L	ND<0.004	ND<0.004
Dibenzo(a,h)anthracene	mg/L	ND<0.004	ND<0.004
Fluoranthene	mg/L	ND<0.004	ND<0.004
Fluorene	mg/L	ND<0.004	ND<0.004
Indeno(1,2,3-cd)pyrene	mg/L	ND<0.004	ND<0.004
Naphthalene	mg/L	ND<0.004	ND<0.004
Phenanthrene	mg/L	ND<0.004	ND<0.004
Pyrene	mg/L	ND<0.004	ND<0.004

Constituent	Units	HV63	JK70
Critical Effluent Dilution	%	0.44	0.39
Date	--	12/20/2019	11/8/2019
Water Quality Parameters (Total)			
Hardness (as CaCO3)	mg/L	5,810	4,560
Alkalinity, Total (As CaCO3)	mg/L	75	52.5
HCO_3^- (Estimated as 1.22 * Total Alk.)	mg/L	91.5	64.1
Total Suspended Solids (Residue, Non-Filterable)	mg/L	10.2	19
Nitrogen, Ammonia (As N)[1]	mg/L	ND<0.5	ND<0.5
Chemical Oxygen Demand	mg/L	ND<300	ND<300
Organic Carbon, Total	mg/L	ND<8	15
Sulfide	mg/L	ND<0.02	0.02
Specific Gravity	@4 °C	1.26	1.03
Water Quality Parameters (Dissolved)			
Total Dissolved Solids (Residue, Filterable)	mg/L	23,900	20,300
Dissolved Organic Carbon	mg/L	ND<2	16.1
Metals (Total)			
As	mg/L	ND<0.01	ND<0.01
Ba	mg/L	0.027	0.026
Cd	mg/L	0.002	0.003
Ca	mg/L	834	282
Cr	mg/L	ND<0.01	ND<0.01
Cu	mg/L	0.006	0.017
Pb	mg/L	ND<0.005	ND<0.005
Mg	mg/L	905	937
Hg	mg/L	0.0000022	0.0000011
Ni	mg/L	ND<0.005	ND<0.005
K	mg/L	277	401
Se	mg/L	0.143	0.148
Na	mg/L	6,930	6,740
Tl	mg/L	ND<0.006	0.01
Zn	mg/L	0.02	0.014
Metals (Dissolved)			
As	mg/L	ND<0.01	0.0139
Ba	mg/L	0.0309	0.0259
Cd	mg/L	0.0016	0.0021
Ca	mg/L	771	284
Cr	mg/L	ND<0.01	ND<0.01
Cu	mg/L	0.0058	0.0132
Pb	mg/L	ND<0.005	ND<0.005
Mg	mg/L	831	929
Hg	mg/L	0.0000016	ND<0.0000005
Ni	mg/L	ND<0.005	ND<0.005
K	mg/L	278	273
Se	mg/L	0.155	0.165
Na	mg/L	6,980	7,440
Tl	mg/L	ND<0.006	0.0065
Zn	mg/L	0.0307	ND<0.01
Inorganic Anions (Total)			
Br	mg/L	38.1	37.8
Cl	mg/L	14,400	13,300
SO_4^{2-}	mg/L	1,900	1,750
Polycyclic Aromatic Hydrocarbons (PAHs)			
Acenaphthene	mg/L	ND<0.004	ND<0.004
Acenaphthylene	mg/L	ND<0.004	ND<0.004
Anthracene	mg/L	ND<0.004	ND<0.004
Benzo(a)anthracene	mg/L	ND<0.004	ND<0.004
Benzo(a)pyrene	mg/L	ND<0.004	ND<0.004
Benzo(b)fluoranthene	mg/L	ND<0.004	ND<0.004
Benzo(g,h,i)perylene	mg/L	ND<0.004	ND<0.004
Benzo(k)fluoranthene	mg/L	ND<0.004	ND<0.004
Chrysene	mg/L	ND<0.004	ND<0.004
Dibenzo(a,h)anthracene	mg/L	ND<0.004	ND<0.004
Fluoranthene	mg/L	ND<0.004	ND<0.004
Fluorene	mg/L	ND<0.004	ND<0.004
Indeno(1,2,3-cd)pyrene	mg/L	ND<0.004	ND<0.004
Naphthalene	mg/L	ND<0.004	ND<0.004
Phenanthrene	mg/L	ND<0.004	ND<0.004
Pyrene	mg/L	ND<0.004	ND<0.004

Constituent	Units	RD67	RU61
Critical Effluent Dilution	%	0.48	0.55
Date	--	11/25/2019	1/6/2020
Water Quality Parameters (Total)			
Hardness (as CaCO3)	mg/L	5,220	5,730
Alkalinity, Total (As CaCO3)	mg/L	77.5	77.5
HCO_3^- (Estimated as 1.22 * Total Alk.)	mg/L	94.6	94.6
Total Suspended Solids (Residue, Non-Filterable)	mg/L	6.6	10.6
Nitrogen, Ammonia (As N)[1]	mg/L	ND<0.5	ND<0.5
Chemical Oxygen Demand	mg/L	ND<300	1,420
Organic Carbon, Total	mg/L	5.2	406
Sulfide	mg/L	0.02	0.021
Specific Gravity	@4 °C	1.24	1.45
Water Quality Parameters (Dissolved)			
Total Dissolved Solids (Residue, Filterable)	mg/L	27,300	29,700
Dissolved Organic Carbon	mg/L	4.34	385
Metals (Total)			
As	mg/L	ND<0.01	ND<0.01
Ba	mg/L	0.043	0.077
Cd	mg/L	0.002	0.002
Ca	mg/L	707	828
Cr	mg/L	ND<0.01	ND<0.01
Cu	mg/L	0.008	0.009
Pb	mg/L	ND<0.005	ND<0.005
Mg	mg/L	839	890
Hg	mg/L	0.0000011	0.0000012
Ni	mg/L	ND<0.005	ND<0.005
K	mg/L	279	286
Se	mg/L	0.165	0.159
Na	mg/L	6,880	6,970
Tl	mg/L	0.008	0.008
Zn	mg/L	0.143	0.092
Metals (Dissolved)			
As	mg/L	ND<0.01	ND<0.01
Ba	mg/L	0.133	0.0402
Cd	mg/L	0.0015	0.0016
Ca	mg/L	701	808
Cr	mg/L	ND<0.01	ND<0.01
Cu	mg/L	0.0117	0.0077
Pb	mg/L	ND<0.005	ND<0.005
Mg	mg/L	833	866
Hg	mg/L	ND<0.0000005	0.000001
Ni	mg/L	ND<0.005	ND<0.005
K	mg/L	290	287
Se	mg/L	0.147	0.161
Na	mg/L	6,900	6,900
Tl	mg/L	ND<0.006	0.0065
Zn	mg/L	0.166	0.0767
Inorganic Anions (Total)			
Br	mg/L	116	2,630
Cl	mg/L	14300	13,200
SO_4^{2-}	mg/L	2020	2,120
Polycyclic Aromatic Hydrocarbons (PAHs)			
Acenaphthene	mg/L	ND<0.004	ND<0.004
Acenaphthylene	mg/L	ND<0.004	ND<0.004
Anthracene	mg/L	ND<0.004	ND<0.004
Benzo(a)anthracene	mg/L	ND<0.004	ND<0.004
Benzo(a)pyrene	mg/L	ND<0.004	ND<0.004
Benzo(b)fluoranthene	mg/L	ND<0.004	ND<0.004
Benzo(g,h,i)perylene	mg/L	ND<0.004	ND<0.004
Benzo(k)fluoranthene	mg/L	ND<0.004	ND<0.004
Chrysene	mg/L	ND<0.004	ND<0.004
Dibenzo(a,h)anthracene	mg/L	ND<0.004	ND<0.004
Fluoranthene	mg/L	ND<0.004	ND<0.004
Fluorene	mg/L	ND<0.004	ND<0.004
Indeno(1,2,3-cd)pyrene	mg/L	ND<0.004	ND<0.004
Naphthalene	mg/L	ND<0.004	ND<0.004
Phenanthrene	mg/L	ND<0.004	ND<0.004
Pyrene	mg/L	XP62	NY50

Table A5. Laboratory Analytical Results for Laboratory Control Seawater and TCW Effluent Samples

Constituent	Units	XP62	NY50
Critical Effluent Dilution	%	0.19	0.39
Date	--	1/23/2020	2/11/2020
Water Quality Parameters (Total)			
Hardness (as CaCO3)	mg/L	4,740	4,620
Alkalinity, Total (As CaCO3)	mg/L	87.5	95
HCO_3^- (Estimated as 1.22 * Total Alk.)	mg/L	106.8	115.9
Total Suspended Solids (Residue, Non-Filterable)	mg/L	7	16.4
Nitrogen, Ammonia (As N)[1]	mg/L	ND<0.5	0.52
Chemical Oxygen Demand	mg/L	ND<150	ND<300
Organic Carbon, Total	mg/L	ND<2	ND<2
Sulfide	mg/L	0.027	0.026
Specific Gravity	@4 °C	1.3	1.12
Water Quality Parameters (Dissolved)			
Total Dissolved Solids (Residue, Filterable)	mg/L	24,900	23,400
Dissolved Organic Carbon	mg/L	ND<2	ND<2
Metals (Total)			
As	mg/L	ND<0.15	ND<0.1
Ba	mg/L	ND<0.1	ND<0.1
Cd	mg/L	ND<0.02	0.01
Ca	mg/L	513	412
Cr	mg/L	ND<0.1	ND<0.1
Cu	mg/L	ND<0.05	ND<0.03
Pb	mg/L	ND<0.05	ND<0.05
Mg	mg/L	839	873
Hg	mg/L	0.0000009	0.0000010
Ni	mg/L	ND<0.05	ND<0.05
K	mg/L	288	274
Se	mg/L	ND<0.3	ND<0.2
Na	mg/L	7,070	7,030
Tl	mg/L	ND<0.06	ND<0.06
Zn	mg/L	ND<0.1	ND<0.1
Metals (Dissolved)			
As	mg/L	ND<0.15	ND<0.1
Ba	mg/L	ND<0.1	ND<0.1
Cd	mg/L	ND<0.02	ND<0.01
Ca	mg/L	505	400
Cr	mg/L	ND<0.1	ND<0.1
Cu	mg/L	ND<0.05	ND<0.05
Pb	mg/L	ND<0.05	ND<0.05
Mg	mg/L	826	863
Hg	mg/L	ND<0.0000005	0.0000008
Ni	mg/L	ND<0.05	ND<0.05
K	mg/L	285	312
Se	mg/L	ND<0.3	ND<0.2
Na	mg/L	6,960	6,990
Tl	mg/L	ND<0.06	ND<0.06
Zn	mg/L	ND<0.1	ND<0.1
Inorganic Anions (Total)			
Br	mg/L	41.7	90.0
Cl	mg/L	14,100	14,100
SO_4^{2-}	mg/L	1,810	2,230
Polycyclic Aromatic Hydrocarbons (PAHs)			
Acenaphthene	mg/L	ND<0.004	ND<0.004
Acenaphthylene	mg/L	ND<0.004	ND<0.004
Anthracene	mg/L	ND<0.004	ND<0.004
Benzo(a)anthracene	mg/L	ND<0.004	ND<0.004
Benzo(a)pyrene	mg/L	ND<0.004	ND<0.004
Benzo(b)fluoranthene	mg/L	ND<0.004	ND<0.004
Benzo(g,h,i)perylene	mg/L	ND<0.004	ND<0.004
Benzo(k)fluoranthene	mg/L	ND<0.004	ND<0.004
Chrysene	mg/L	ND<0.004	ND<0.004
Dibenzo(a,h)anthracene	mg/L	ND<0.004	ND<0.004
Fluoranthene	mg/L	ND<0.004	ND<0.004
Fluorene	mg/L	ND<0.004	ND<0.004
Indeno(1,2,3-cd)pyrene	mg/L	ND<0.004	ND<0.004
Naphthalene	mg/L	ND<0.004	ND<0.004
Phenanthrene	mg/L	ND<0.004	ND<0.004
Pyrene	mg/L	ND<0.004	ND<0.004

Table A5. Laboratory Analytical Results for Laboratory Control Seawater and TCW Effluent Samples

Constituent	Units	LC54	AU71
Critical Effluent Dilution	%	1.25	0.39
Date	--	2/18/2020	3/18/2020
Water Quality Parameters (Total)			
Hardness (as CaCO3)	mg/L	4,920	4,340
Alkalinity, Total (As CaCO3)	mg/L	97.5	70
HCO_3^- (Estimated as 1.22 * Total Alk.)	mg/L	119.0	85.4
Total Suspended Solids (Residue, Non-Filterable)	mg/L	ND<5	18.6
Nitrogen, Ammonia (As N)[1]	mg/L	ND<0.5	ND<0.5
Chemical Oxygen Demand	mg/L	ND<150	ND<300
Organic Carbon, Total	mg/L	12.1	7.7
Sulfide	mg/L	0.031	0.023
Specific Gravity	@4 °C	1.06	1.15
Water Quality Parameters (Dissolved)			
Total Dissolved Solids (Residue, Filterable)	mg/L	26,000	23,500
Dissolved Organic Carbon	mg/L	7.8	7.48
Metals (Total)			
As	mg/L	0.111	ND<0.1
Ba	mg/L	ND<0.1	ND<0.1
Cd	mg/L	ND<0.01	ND<0.01
Ca	mg/L	429	276
Cr	mg/L	ND<0.1	ND<0.1
Cu	mg/L	0.035	ND<0.03
Pb	mg/L	ND<0.05	ND<0.05
Mg	mg/L	935	887
Hg	mg/L	0.0000009	0.0000015
Ni	mg/L	ND<0.05	ND<0.05
K	mg/L	381	381
Se	mg/L	0.344	ND<0.2
Na	mg/L	7690	7450
Tl	mg/L	ND<0.06	ND<0.06
Zn	mg/L	0.105	ND<0.1
Metals (Dissolved)			
As	mg/L	ND<0.1	ND<0.1
Ba	mg/L	ND<0.1	ND<0.1
Cd	mg/L	ND<0.01	ND<0.01
Ca	mg/L	388	267
Cr	mg/L	ND<0.1	ND<0.1
Cu	mg/L	ND<0.05	ND<0.05
Pb	mg/L	ND<0.05	ND<0.05
Mg	mg/L	857	857
Hg	mg/L	0.000001	0.0000015
Ni	mg/L	ND<0.05	ND<0.05
K	mg/L	312	373
Se	mg/L	0.208	ND<0.4
Na	mg/L	7020	7260
Tl	mg/L	ND<0.06	0.085
Zn	mg/L	ND<0.1	ND<0.1
Inorganic Anions (Total)			
Br	mg/L	59.7	44.1
Cl	mg/L	15,700	15,400
SO_4^{2-}	mg/L	2,140	2,100
Polycyclic Aromatic Hydrocarbons (PAHs)			
Acenaphthene	mg/L	ND<0.004	ND<0.004
Acenaphthylene	mg/L	ND<0.004	ND<0.004
Anthracene	mg/L	ND<0.004	ND<0.004
Benzo(a)anthracene	mg/L	ND<0.004	ND<0.004
Benzo(a)pyrene	mg/L	ND<0.004	ND<0.004
Benzo(b)fluoranthene	mg/L	ND<0.004	ND<0.004
Benzo(g,h,i)perylene	mg/L	ND<0.004	ND<0.004
Benzo(k)fluoranthene	mg/L	ND<0.004	ND<0.004
Chrysene	mg/L	ND<0.004	ND<0.004
Dibenzo(a,h)anthracene	mg/L	ND<0.004	ND<0.004
Fluoranthene	mg/L	ND<0.004	ND<0.004
Fluorene	mg/L	ND<0.004	ND<0.004
Indeno(1,2,3-cd)pyrene	mg/L	ND<0.004	ND<0.004
Naphthalene	mg/L	ND<0.004	ND<0.004
Phenanthrene	mg/L	ND<0.004	ND<0.004
Pyrene	mg/L	ND<0.004	ND<0.004

Constituent	Units	YO64	FP89
Critical Effluent Dilution	%	0.39	0.39
Date	--	5/12/2020	3/18/2020
Water Quality Parameters (Total)			
Hardness (as CaCO3)	mg/L	3,040	4,190
Alkalinity, Total (As CaCO3)	mg/L	90	77.5
HCO_3^- (Estimated as 1.22 * Total Alk.)	mg/L	109.8	94.6
Total Suspended Solids (Residue, Non-Filterable)	mg/L	76.6	17.8
Nitrogen, Ammonia (As N)[1]	mg/L	ND<0.5	ND<0.5
Chemical Oxygen Demand	mg/L	580	ND<300
Organic Carbon, Total	mg/L	70.3	9
Sulfide	mg/L	ND<0.02	0.02
Specific Gravity	@4 °C	[See Note 1]	1.04
Water Quality Parameters (Dissolved)			
Total Dissolved Solids (Residue, Filterable)	mg/L	26,900	24,400
Dissolved Organic Carbon	mg/L	126	9.14
Metals (Total)			
As	mg/L	ND<0.1	ND<0.1
Ba	mg/L	ND<0.1	ND<0.1
Cd	mg/L	ND<0.01	ND<0.01
Ca	mg/L	220	261
Cr	mg/L	ND<0.1	ND<0.1
Cu	mg/L	0.034	ND<0.03
Pb	mg/L	ND<0.05	ND<0.05
Mg	mg/L	604	858
Hg	mg/L	0.0000017	0.0000012
Ni	mg/L	ND<0.05	ND<0.05
K	mg/L	201	367
Se	mg/L	ND<0.2	ND<0.2
Na	mg/L	4830	6970
Tl	mg/L	ND<0.06	ND<0.06
Zn	mg/L	0.226	ND<0.1
Metals (Dissolved)			
As	mg/L	ND<0.1	ND<0.1
Ba	mg/L	ND<0.1	ND<0.1
Cd	mg/L	ND<0.01	ND<0.01
Ca	mg/L	307	260
Cr	mg/L	ND<0.1	ND<0.1
Cu	mg/L	ND<0.05	ND<0.05
Pb	mg/L	ND<0.05	ND<0.05
Mg	mg/L	844	853
Hg	mg/L	0.0000016	0.0000010
Ni	mg/L	ND<0.05	ND<0.05
K	mg/L	298	368
Se	mg/L	0.341	ND<0.4
Na	mg/L	6660	6990
Tl	mg/L	ND<0.06	ND<0.06
Zn	mg/L	0.356	ND<0.1
Inorganic Anions (Total)			
Br	mg/L	263	37.8
Cl	mg/L	13,000	14,500
SO_4^{2-}	mg/L	1,860	2,140
Polycyclic Aromatic Hydrocarbons (PAHs)			
Acenaphthene	mg/L	ND<0.004	ND<0.004
Acenaphthylene	mg/L	ND<0.004	ND<0.004
Anthracene	mg/L	ND<0.004	ND<0.004
Benzo(a)anthracene	mg/L	ND<0.004	ND<0.004
Benzo(a)pyrene	mg/L	ND<0.004	ND<0.004
Benzo(b)fluoranthene	mg/L	ND<0.004	ND<0.004
Benzo(g,h,i)perylene	mg/L	ND<0.004	ND<0.004
Benzo(k)fluoranthene	mg/L	ND<0.004	ND<0.004
Chrysene	mg/L	ND<0.004	ND<0.004
Dibenzo(a,h)anthracene	mg/L	ND<0.004	ND<0.004
Fluoranthene	mg/L	ND<0.004	ND<0.004
Fluorene	mg/L	ND<0.004	ND<0.004
Indeno(1,2,3-cd)pyrene	mg/L	ND<0.004	ND<0.004
Naphthalene	mg/L	ND<0.004	ND<0.004
Phenanthrene	mg/L	ND<0.004	ND<0.004
Pyrene	mg/L	ND<0.004	ND<0.004

Constituent	Units	ZG57	GQ67
Critical Effluent Dilution	%	0.291	0.1
Date	--	3/2/2020	5/14/2020
Water Quality Parameters (Total)			
Hardness (as CaCO3)	mg/L	3,980	4,630
Alkalinity, Total (As CaCO3)	mg/L	77.5	77.5
HCO_3^- (Estimated as 1.22 * Total Alk.)	mg/L	94.6	94.6
Total Suspended Solids (Residue, Non-Filterable)	mg/L	ND<5	ND<5
Nitrogen, Ammonia (As N)[1]	mg/L	ND<0.5	ND<0.5
Chemical Oxygen Demand	mg/L	ND<300	ND<300
Organic Carbon, Total	mg/L	ND<4	2.7
Sulfide	mg/L	0.028	ND<0.02
Specific Gravity	@4 °C	1.02	1.49
Water Quality Parameters (Dissolved)			
Total Dissolved Solids (Residue, Filterable)	mg/L	23,700	24,800
Dissolved Organic Carbon	mg/L	ND<2	ND<2
Metals (Total)			
As	mg/L	ND<0.1	ND<0.1
Ba	mg/L	ND<0.1	ND<0.1
Cd	mg/L	ND<0.01	ND<0.01
Ca	mg/L	251	387
Cr	mg/L	ND<0.1	ND<0.1
Cu	mg/L	ND<0.03	ND<0.03
Pb	mg/L	ND<0.05	ND<0.05
Mg	mg/L	814	889
Hg	mg/L	0.0000014	0.0000010
Ni	mg/L	ND<0.05	ND<0.05
K	mg/L	272	417
Se	mg/L	0.461	0
Na	mg/L	6410	6,920
Tl	mg/L	ND<0.06	ND<0.1
Zn	mg/L	ND<0.1	ND<0.1
Metals (Dissolved)			
As	mg/L	ND<0.1	ND<0.1
Ba	mg/L	ND<0.1	ND<0.1
Cd	mg/L	ND<0.01	ND<0.01
Ca	mg/L	253	372
Cr	mg/L	ND<0.1	ND<0.1
Cu	mg/L	ND<0.03	ND<0.05
Pb	mg/L	ND<0.05	ND<0.05
Mg	mg/L	828	852
Hg	mg/L	0.0000012	0.0000011
Ni	mg/L	ND<0.05	ND<0.05
K	mg/L	276	300
Se	mg/L	0.359	0.283
Na	mg/L	6540	6,920
Tl	mg/L	ND<0.1	ND<0.06
Zn	mg/L	ND<0.1	ND<0.1
Inorganic Anions (Total)			
Br	mg/L	37.5	490
Cl	mg/L	13,800	14,000
SO_4^{2-}	mg/L	1,990	1,940
Polycyclic Aromatic Hydrocarbons (PAHs)			
Acenaphthene	mg/L	ND<0.004	ND<0.004
Acenaphthylene	mg/L	ND<0.004	ND<0.004
Anthracene	mg/L	ND<0.004	ND<0.004
Benzo(a)anthracene	mg/L	ND<0.004	ND<0.004
Benzo(a)pyrene	mg/L	ND<0.004	ND<0.004
Benzo(b)fluoranthene	mg/L	ND<0.004	ND<0.004
Benzo(g,h,i)perylene	mg/L	ND<0.004	ND<0.004
Benzo(k)fluoranthene	mg/L	ND<0.004	ND<0.004
Chrysene	mg/L	ND<0.004	ND<0.004
Dibenzo(a,h)anthracene	mg/L	ND<0.004	ND<0.004
Fluoranthene	mg/L	ND<0.004	ND<0.004
Fluorene	mg/L	ND<0.004	ND<0.004
Indeno(1,2,3-cd)pyrene	mg/L	ND<0.004	ND<0.004
Naphthalene	mg/L	ND<0.004	ND<0.004
Phenanthrene	mg/L	ND<0.004	ND<0.004
Pyrene	mg/L	ND<0.004	ND<0.004

Table A5. Laboratory Analytical Results for Laboratory Control Seawater and TCW Effluent Samples

Constituent	Units	YU91	LX98
Critical Effluent Dilution	%	0.41	0.56
Date	--	5/28/2020	5/8/2020
Water Quality Parameters (Total)			
Hardness (as CaCO3)	mg/L	3,670	3,870
Alkalinity, Total (As CaCO3)	mg/L	72.5	75
HCO_3^- (Estimated as 1.22 * Total Alk.)	mg/L	88.5	73.8
Total Suspended Solids (Residue, Non-Filterable)	mg/L	22.8	15.4
Nitrogen, Ammonia (As N)[1]	mg/L	ND<0.50	ND<0.5
Chemical Oxygen Demand	mg/L	ND<300	ND<300
Organic Carbon, Total	mg/L	6	ND<2
Sulfide	mg/L	0.02	ND<0.02
Specific Gravity	@4 °C	[See Note 1]	1.01
Water Quality Parameters (Dissolved)			
Total Dissolved Solids (Residue, Filterable)	mg/L	24,000	24,600
Dissolved Organic Carbon	mg/L	6.12	ND<2
Metals (Total)			
As	mg/L	ND<0.1	ND<0.1
Ba	mg/L	ND<0.1	BD<0.1
Cd	mg/L	ND<0.05	ND<0.01
Ca	mg/L	234	237
Cr	mg/L	ND<0.1	ND<0.1
Cu	mg/L	0.044	ND<0.03
Pb	mg/L	ND<0.050	ND<0.05
Mg	mg/L	749	796
Hg	mg/L	0.0000013	0.0000013
Ni	mg/L	ND<0.50	ND<0.05
K	mg/L	247	499
Se	mg/L	0.473	0.314
Na	mg/L	5,810	6,280
Tl	mg/L	ND<0.06	ND<0.06
Zn	mg/L	ND<0.1	ND<0.1
Metals (Dissolved)			
As	mg/L	ND<0.1	ND<0.1
Ba	mg/L	ND<0.1	ND<0.1
Cd	mg/L	<0.05	ND<0.01
Ca	mg/L	282	221
Cr	mg/L	ND<0.100	ND<0.1
Cu	mg/L	ND<0.05	ND<0.05
Pb	mg/L	ND<0.05	ND<0.05
Mg	mg/L	906	746
Hg	mg/L	0.0000010	0.0000014
Ni	mg/L	ND<0.05	ND<0.05
K	mg/L	301	504
Se	mg/L	0.445	0.272
Na	mg/L	7,020	5,890
Tl	mg/L	ND<0.06	ND<0.06
Zn	mg/L	ND<0.1	ND<0.1
Inorganic Anions (Total)			
Br	mg/L	54.7	39.5
Cl	mg/L	14,100	13,800
SO_4^{2-}	mg/L	1,980	1,880
Polycyclic Aromatic Hydrocarbons (PAHs)			
Acenaphthene	mg/L	ND<0.004	ND<0.004
Acenaphthylene	mg/L	ND<0.004	ND<0.004
Anthracene	mg/L	ND<0.004	ND<0.004
Benzo(a)anthracene	mg/L	ND<0.004	ND<0.004
Benzo(a)pyrene	mg/L	ND<0.004	ND<0.004
Benzo(b)fluoranthene	mg/L	ND<0.004	ND<0.004
Benzo(g,h,i)perylene	mg/L	ND<0.004	ND<0.004
Benzo(k)fluoranthene	mg/L	ND<0.004	ND<0.004
Chrysene	mg/L	ND<0.004	ND<0.004
Dibenzo(a,h)anthracene	mg/L	ND<0.004	ND<0.004
Fluoranthene	mg/L	ND<0.004	ND<0.004
Fluorene	mg/L	ND<0.004	ND<0.004
Indeno(1,2,3-cd)pyrene	mg/L	ND<0.004	ND<0.004
Naphthalene	mg/L	ND<0.004	ND<0.004
Phenanthrene	mg/L	ND<0.004	ND<0.004
Pyrene	mg/L	ND<0.004	ND<0.004

Table A5. Laboratory Analytical Results for Laboratory Control Seawater and TCW Effluent Samples

Constituent	Units	IS88	RU72
Critical Effluent Dilution	%	0.65	0.36
Date	--	5/28/2020	5/22/2020
Water Quality Parameters (Total)			
Hardness (as CaCO3)	mg/L	4,140	4,240
Alkalinity, Total (As CaCO3)	mg/L	72.5	105
HCO_3^- (Estimated as 1.22 * Total Alk.)	mg/L	88.5	128.1
Total Suspended Solids (Residue, Non-Filterable)	mg/L	14	19
Nitrogen, Ammonia (As N)[1]	mg/L	ND<0.5	ND<0.5
Chemical Oxygen Demand	mg/L	ND<300	ND<300
Organic Carbon, Total	mg/L	ND<2	16.2
Sulfide	mg/L	0.026	ND<0.02
Specific Gravity	@4 °C	1.02	[See Note 1]
Water Quality Parameters (Dissolved)			
Total Dissolved Solids (Residue, Filterable)	mg/L	25,400	32,900
Dissolved Organic Carbon	mg/L	ND<2	16.5
Metals (Total)			
As	mg/L	ND<0.1	ND<0.1
Ba	mg/L	ND<0.1	ND<0.1
Cd	mg/L	ND<0.01	ND<0.01
Ca	mg/L	254	263
Cr	mg/L	ND<0.1	ND<0.1
Cu	mg/L	0.037	0.034
Pb	mg/L	ND<0.05	ND<0.05
Mg	mg/L	851	870
Hg	mg/L	0.0000027	0.0000011
Ni	mg/L	ND<0.05	ND<0.05
K	mg/L	417	391
Se	mg/L	ND<0.2	0.234
Na	mg/L	6,730	6,840
Tl	mg/L	0.092	ND<0.06
Zn	mg/L	0.152	ND<0.1
Metals (Dissolved)			
As	mg/L	ND<0.1	0.288
Ba	mg/L	ND<0.1	ND<0.1
Cd	mg/L	ND<0.01	0.013
Ca	mg/L	216	284
Cr	mg/L	ND<0.1	ND<0.1
Cu	mg/L	ND<0.05	ND<0.05
Pb	mg/L	ND<0.05	ND<0.05
Mg	mg/L	735	927
Hg	mg/L	0.0000023	0.0000012
Ni	mg/L	ND<0.05	ND<0.05
K	mg/L	445	430
Se	mg/L	0.381	0.465
Na	mg/L	5,840	8,310
Tl	mg/L	0.119	ND<0.06
Zn	mg/L	ND<0.1	ND<0.1
Inorganic Anions (Total)			
Br	mg/L	38.7	49
Cl	mg/L	13,800	13,900
SO_4^{2-}	mg/L	1,980	1,790
Polycyclic Aromatic Hydrocarbons (PAHs)			
Acenaphthene	mg/L	ND<0.004	ND<0.004
Acenaphthylene	mg/L	ND<0.004	ND<0.004
Anthracene	mg/L	ND<0.004	ND<0.004
Benzo(a)anthracene	mg/L	ND<0.004	ND<0.004
Benzo(a)pyrene	mg/L	ND<0.004	ND<0.004
Benzo(b)fluoranthene	mg/L	ND<0.004	ND<0.004
Benzo(g,h,i)perylene	mg/L	ND<0.004	ND<0.004
Benzo(k)fluoranthene	mg/L	ND<0.004	ND<0.004
Chrysene	mg/L	ND<0.004	ND<0.004
Dibenzo(a,h)anthracene	mg/L	ND<0.004	ND<0.004
Fluoranthene	mg/L	ND<0.004	ND<0.004
Fluorene	mg/L	ND<0.004	ND<0.004
Indeno(1,2,3-cd)pyrene	mg/L	ND<0.004	ND<0.004
Naphthalene	mg/L	ND<0.004	ND<0.004
Phenanthrene	mg/L	ND<0.004	ND<0.004
Pyrene	mg/L	ND<0.004	ND<0.004

Constituent	Units	IH80[2]	BT52[3]
Critical Effluent Dilution	%	Sample not analyzed.	Sample not analyzed.
Date	--		
Water Quality Parameters (Total)			
Hardness (as CaCO3)	mg/L	Sample not analyzed.	Sample not analyzed.
Alkalinity, Total (As CaCO3)	mg/L		
HCO_3^- (Estimated as 1.22 * Total Alk.)	mg/L		
Total Suspended Solids (Residue, Non-Filterable)	mg/L		
Nitrogen, Ammonia (As N)[1]	mg/L		
Chemical Oxygen Demand	mg/L		
Organic Carbon, Total	mg/L		
Sulfide	mg/L		
Specific Gravity	@4 °C		
Water Quality Parameters (Dissolved)			
Total Dissolved Solids (Residue, Filterable)	mg/L	Sample not analyzed.	Sample not analyzed.
Dissolved Organic Carbon	mg/L		
Metals (Total)			
As	mg/L	Sample not analyzed	Sample not analyzed.
Ba	mg/L		
Cd	mg/L		
Ca	mg/L		
Cr	mg/L		
Cu	mg/L		
Pb	mg/L		
Mg	mg/L		
Hg	mg/L		
Ni	mg/L		
K	mg/L		
Se	mg/L		
Na	mg/L		
Tl	mg/L		
Zn	mg/L		
Metals (Dissolved)			
As	mg/L	Sample not analyzed.	Sample not analyzed.
Ba	mg/L		
Cd	mg/L		
Ca	mg/L		
Cr	mg/L		
Cu	mg/L		
Pb	mg/L		
Mg	mg/L		
Hg	mg/L		
Ni	mg/L		
K	mg/L		
Se	mg/L		
Na	mg/L		
Tl	mg/L		
Zn	mg/L		
Inorganic Anions (Total)			
Br	mg/L	Sample not analyzed.	Sample not analyzed.
Cl	mg/L		
SO_4^{2-}	mg/L		
Polycyclic Aromatic Hydrocarbons (PAHs)			
Acenaphthene	mg/L	Sample not analyzed.	Sample not analyzed.
Acenaphthylene	mg/L		
Anthracene	mg/L		
Benzo(a)anthracene	mg/L		
Benzo(a)pyrene	mg/L		
Benzo(b)fluoranthene	mg/L		
Benzo(g,h,i)perylene	mg/L		
Benzo(k)fluoranthene	mg/L		
Chrysene	mg/L		
Dibenzo(a,h)anthracene	mg/L		
Fluoranthene	mg/L		
Fluorene	mg/L		
Indeno(1,2,3-cd)pyrene	mg/L		
Naphthalene	mg/L		
Phenanthrene	mg/L		
Pyrene	mg/L		

Table A5. Laboratory Analytical Results for Laboratory Control Seawater and TCW Effluent Samples

Constituent	Units	SH87	EP57
Critical Effluent Dilution	%	0.33	0.16
Date	--	5/12/2020	5/10/2020
Water Quality Parameters (Total)			
Hardness (as CaCO3)	mg/L	4310	4490
Alkalinity, Total (As CaCO3)	mg/L	80	82.5
HCO_3^- (Estimated as 1.22 * Total Alk.)	mg/L	97.6	100.7
Total Suspended Solids (Residue, Non-Filterable)	mg/L	9	12.2
Nitrogen, Ammonia (As N)[1]	mg/L	ND<0.5	ND<0.5
Chemical Oxygen Demand	mg/L	ND<300	ND<300
Organic Carbon, Total	mg/L	ND<2	ND<2
Sulfide	mg/L	ND<0.02	ND<0.02
Specific Gravity	@4 °C	1.05	1.05
Water Quality Parameters (Dissolved)			
Total Dissolved Solids (Residue, Filterable)	mg/L	23,700	20,300
Dissolved Organic Carbon	mg/L	ND<2	ND<2
Metals (Total)			
As	mg/L	0.181	ND<0.1
Ba	mg/L	ND<0.1	ND<0.1
Cd	mg/L	0.01	ND<0.01
Ca	mg/L	265	280
Cr	mg/L	ND<0.1	ND<0.1
Cu	mg/L	0.038	0.046
Pb	mg/L	ND<0.05	ND<0.05
Mg	mg/L	886	922
Hg	mg/L	0.0000013	0.0000022
Ni	mg/L	ND<0.05	ND<0.05
K	mg/L	310	306
Se	mg/L	0.352	0.369
Na	mg/L	6990	7080
Tl	mg/L	ND<0.06	ND<0.06
Zn	mg/L	ND<0.1	ND<0.1
Metals (Dissolved)			
As	mg/L	ND<0.1	ND<0.1
Ba	mg/L	ND<0.1	ND<0.1
Cd	mg/L	ND<0.01	ND<0.01
Ca	mg/L	267	273
Cr	mg/L	ND<0.1	ND<0.1
Cu	mg/L	ND<0.05	0.046
Pb	mg/L	ND<0.05	ND<0.05
Mg	mg/L	895	901
Hg	mg/L	0.0000008	0.0000005
Ni	mg/L	ND<0.05	ND<0.05
K	mg/L	310	309
Se	mg/L	0.352	0.282
Na	mg/L	6990	6970
Tl	mg/L	ND<0.06	ND<0.06
Zn	mg/L	ND<0.1	ND<0.1
Inorganic Anions (Total)			
Br	mg/L	44.1	54.9
Cl	mg/L	13600	13300
SO_4^{2-}	mg/L	1830	1850
Polycyclic Aromatic Hydrocarbons (PAHs)			
Acenaphthene	mg/L	ND<0.004	ND<0.004
Acenaphthylene	mg/L	ND<0.004	ND<0.004
Anthracene	mg/L	ND<0.004	ND<0.004
Benzo(a)anthracene	mg/L	ND<0.004	ND<0.004
Benzo(a)pyrene	mg/L	ND<0.004	ND<0.004
Benzo(b)fluoranthene	mg/L	ND<0.004	ND<0.004
Benzo(g,h,i)perylene	mg/L	ND<0.004	ND<0.004
Benzo(k)fluoranthene	mg/L	ND<0.004	ND<0.004
Chrysene	mg/L	ND<0.004	ND<0.004
Dibenzo(a,h)anthracene	mg/L	ND<0.004	ND<0.004
Fluoranthene	mg/L	ND<0.004	ND<0.004
Fluorene	mg/L	ND<0.004	ND<0.004
Indeno(1,2,3-cd)pyrene	mg/L	ND<0.004	ND<0.004
Naphthalene	mg/L	ND<0.004	ND<0.004
Phenanthrene	mg/L	ND<0.004	ND<0.004
Pyrene	mg/L	ND<0.004	ND<0.004

Table A5. Laboratory Analytical Results for Laboratory Control Seawater and TCW Effluent Samples

Constituent	Units	TR84	RC74
Critical Effluent Dilution	%	0.21	0.05
Date	--	5/12/2020	5/24/2020
Water Quality Parameters (Total)			
Hardness (as CaCO3)	mg/L	922	4830
Alkalinity, Total (As CaCO3)	mg/L	80	72.5
HCO_3^- (Estimated as 1.22 * Total Alk.)	mg/L	97.6	88.5
Total Suspended Solids (Residue, Non-Filterable)	mg/L	17	18.6
Nitrogen, Ammonia (As N)[1]	mg/L	ND<0.5	ND<0.5
Chemical Oxygen Demand	mg/L	ND<300	ND<300
Organic Carbon, Total	mg/L	ND<2	ND<40
Sulfide	mg/L	ND<0.02	ND<0.02
Specific Gravity	@4 °C	1.06	1.01
Water Quality Parameters (Dissolved)			
Total Dissolved Solids (Residue, Filterable)	mg/L	21,800	25,200
Dissolved Organic Carbon	mg/L	ND<2	ND<40
Metals (Total)			
As	mg/L	ND<0.1	ND<0.1
Ba	mg/L	ND<0.1	ND<0.1
Cd	mg/L	0.01	ND<0.01
Ca	mg/L	285	296
Cr	mg/L	ND<0.1	ND<0.1
Cu	mg/L	0.046	ND<0.03
Pb	mg/L	ND<0.05	ND<0.05
Mg	mg/L	922	993
Hg	mg/L	0.0000014	0.0000011
Ni	mg/L	ND<0.05	ND<0.05
K	mg/L	305	302
Se	mg/L	0.327	ND<0.2
Na	mg/L	7,070	7,640
Tl	mg/L	ND<0.06	ND<0.06
Zn	mg/L	ND<0.1	ND<0.1
Metals (Dissolved)			
As	mg/L	ND<0.1	ND<0.1
Ba	mg/L	ND<0.1	ND<0.1
Cd	mg/L	ND<0.01	ND<0.01
Ca	mg/L	271	274
Cr	mg/L	ND<0.1	ND<0.1
Cu	mg/L	ND<0.05	ND<0.05
Pb	mg/L	ND<0.05	ND<0.05
Mg	mg/L	878	922
Hg	mg/L	0.0000005	0.0000005
Ni	mg/L	ND<0.05	ND<0.05
K	mg/L	299	285
Se	mg/L	0.369	ND<0.2
Na	mg/L	6790	7110
Tl	mg/L	ND<0.06	ND<0.1
Zn	mg/L	ND<0.1	ND<0.1
Inorganic Anions (Total)			
Br	mg/L	77	38.8
Cl	mg/L	13,300	13,900
SO_4^{2-}	mg/L	1,620	1,890
Polycyclic Aromatic Hydrocarbons (PAHs)			
Acenaphthene	mg/L	ND<0.004	ND<0.004
Acenaphthylene	mg/L	ND<0.004	ND<0.004
Anthracene	mg/L	ND<0.004	ND<0.004
Benzo(a)anthracene	mg/L	ND<0.004	ND<0.004
Benzo(a)pyrene	mg/L	ND<0.004	ND<0.004
Benzo(b)fluoranthene	mg/L	ND<0.004	ND<0.004
Benzo(g,h,i)perylene	mg/L	ND<0.004	ND<0.004
Benzo(k)fluoranthene	mg/L	ND<0.004	ND<0.004
Chrysene	mg/L	ND<0.004	ND<0.004
Dibenzo(a,h)anthracene	mg/L	ND<0.004	ND<0.004
Fluoranthene	mg/L	ND<0.004	ND<0.004
Fluorene	mg/L	ND<0.004	ND<0.004
Indeno(1,2,3-cd)pyrene	mg/L	ND<0.004	ND<0.004
Naphthalene	mg/L	ND<0.004	ND<0.004
Phenanthrene	mg/L	ND<0.004	ND<0.004
Pyrene	mg/L	ND<0.004	ND<0.004

Table A5. Laboratory Analytical Results for Laboratory Control Seawater and TCW Effluent Samples

Constituent	Units	OD76	TF74
Critical Effluent Dilution	%	0.39	0.56
Date	--	5/25/2020	5/25/2020
Water Quality Parameters (Total)			
Hardness (as CaCO3)	mg/L	4180	9720
Alkalinity, Total (As CaCO3)	mg/L	75	77.5
HCO_3^- (Estimated as 1.22 * Total Alk.)	mg/L	91.5	94.6
Total Suspended Solids (Residue, Non-Filterable)	mg/L	16	27
Nitrogen, Ammonia (As N)[1]	mg/L	ND<0.5	ND<0.5
Chemical Oxygen Demand	mg/L	ND<300	960
Organic Carbon, Total	mg/L	ND<40	41.3
Sulfide	mg/L	ND<0.02	ND<0.02
Specific Gravity	@4 °C	[See Note 1]	1.66
Water Quality Parameters (Dissolved)			
Total Dissolved Solids (Residue, Filterable)	mg/L	25,800	39,400
Dissolved Organic Carbon	mg/L	ND<40	43.9
Metals (Total)			
As	mg/L	ND<0.1	ND<0.1
Ba	mg/L	ND<0.1	0.135
Cd	mg/L	ND<0.01	ND<0.01
Ca	mg/L	267	2370
Cr	mg/L	ND<0.1	ND<0.1
Cu	mg/L	ND<0.03	0.055
Pb	mg/L	ND<0.05	ND<0.05
Mg	mg/L	853	926
Hg	mg/L	0.0000068	0.0000017
Ni	mg/L	ND<0.05	ND<0.05
K	mg/L	296	291
Se	mg/L	0.337	0.218
Na	mg/L	6,960	7,260
Tl	mg/L	ND<0.06	ND<0.06
Zn	mg/L	ND<0.1	ND<0.1
Metals (Dissolved)			
As	mg/L	ND<0.1	ND<0.1
Ba	mg/L	ND<0.1	0.138
Cd	mg/L	ND<0.01	ND<0.01
Ca	mg/L	313	2140
Cr	mg/L	ND<0.1	ND<0.1
Cu	mg/L	ND<0.05	ND<0.05
Pb	mg/L	ND<0.05	ND<0.05
Mg	mg/L	1030	839
Hg	mg/L	0.0000021	0.0000008
Ni	mg/L	ND<0.05	ND<0.05
K	mg/L	290	271
Se	mg/L	0.389	0.317
Na	mg/L	7890	6550
Tl	mg/L	ND<0.06	ND<0.1
Zn	mg/L	ND<0.1	ND<0.1
Inorganic Anions (Total)			
Br	mg/L	36.7	8,850
Cl	mg/L	13,800	13,700
SO_4^{2-}	mg/L	2,000	1,880
Polycyclic Aromatic Hydrocarbons (PAHs)			
Acenaphthene	mg/L	ND<0.004	ND<0.004
Acenaphthylene	mg/L	ND<0.004	ND<0.004
Anthracene	mg/L	ND<0.004	ND<0.004
Benzo(a)anthracene	mg/L	ND<0.004	ND<0.004
Benzo(a)pyrene	mg/L	ND<0.004	ND<0.004
Benzo(b)fluoranthene	mg/L	ND<0.004	ND<0.004
Benzo(g,h,i)perylene	mg/L	ND<0.004	ND<0.004
Benzo(k)fluoranthene	mg/L	ND<0.004	ND<0.004
Chrysene	mg/L	ND<0.004	ND<0.004
Dibenzo(a,h)anthracene	mg/L	ND<0.004	ND<0.004
Fluoranthene	mg/L	ND<0.004	ND<0.004
Fluorene	mg/L	ND<0.004	ND<0.004
Indeno(1,2,3-cd)pyrene	mg/L	ND<0.004	ND<0.004
Naphthalene	mg/L	ND<0.004	ND<0.004
Phenanthrene	mg/L	ND<0.004	ND<0.004
Pyrene	mg/L	ND<0.004	ND<0.004

Notes:
mg/L; milligrams per liter
%; percent
°C; degrees Celcius
ND; not detected above the laboratory reporting limit

[1] Due to their viscosity, the analysis of specific gravity was not conducted on Category III gel samples. Also, TCW Category III sample RU72 had insufficient sample volume due to the presence of proppant.
[2] IH80; sample contained a separate phase. Sample was not discharged.
[3] BT52; insufficient sample volume was collected in the field.

Table A6. GHS Acute Aquatic Toxicity
Classification of Chemical Products in TCW Category I Effluents

TCW Sample	Chemical Product Code	GHS Acute Aquatic Toxicity Category Classification	Notes
HV63	No chemical additives	--	--
RD67	Defoamer 1	Acute 2	Identified in SDS Section 2.
	Viscosifier 1	Not Assessed	Identified as Not Classified in SDS Section 2. Contains no hazardous substances in concentrations above cut-off values according to the competent authority. No ecological data.
	Non-emulsifier 1	Acute 2	Identified in SDS Section 2.
RU61	Acid 2	Not Assessed	No GHS classification provided in Section 2. Contains 30-60% acetic acid. 48-h EC50 = 65 mg/L (Daphnia magna). Effect concentrations in the aquatic environment are attributable to a change in pH.
	Acid 5	Not Assessed	No GHS classification provided in Section 2. Contains 30-60% hydrochloric acid. LC50s for fish range from 20.5 - 282 mg/L; LC50 for pH (3.25-3.5). 48-h EC50 for Daphnia magna is 4.92 mg/L.
	Non-emulsifier 1	Acute 2	Identified in SDS Section 2.
XP62	No chemical additives	--	--
LC54	Biocide 1	Acute 1	Identified in SDS Section 2.
	Non-emulsifier 1	Acute 2	Identified in SDS Section 2.
AU71	Well cleaner 1	Not Assessed	No GHS classification provided in Section 2. Manufacturer product toxicity data provided in SDS Section 12 reports product data of: Algae Toxicity EC50 (72h) >10 mg/L (Pseudokirchneriella subcapitata). Acute Crustaceans Toxicity: EC50 (48h) >10 mg/L (Daphnia magna).
	pH Control 3	Not Assessed	No GHS classification for aquatic toxicity identified in Section 2 or for individual substances in Section 3. Product contains sodium hydroxide (10-30%); no other substances identified. 24/48/96-h LC50s for fish range from 125-189 mg/L; 48-h EC50 for Ceriodaphnia sp. Is 40.4 mg/L.
	Non-emulsifier 1	Acute 2	Identified in SDS Section 2.
	Viscosifier 2	Not Assessed	No GHS classification provided in Section 2. No GHS classification for individual substances (dipropylene glycol monomethyl ether; 30-60%) provided in Section 3. SDS indicates in Section 12 that the product is not classified as hazardous to the environment. A NOEC of 0.5 mg/L (*Daphnia magna*) was identified in the SDS for dipropylene glycol monomethyl ether.
	Biocide 4	Biocide 4	Identified in SDS Section 2.

Table A6. GHS Acute Aquatic Toxicity
Classification of Chemical Products in TCW Category I Effluents

TCW Sample	Product	GHS Acute Aquatic Toxicity Category Classification	Notes
ZG57	Defoamer 2	Not Assessed	No GHS classification provided in Section 2. Product consists of kerosene (60-100%); naphthalene (1-5%); and ethylbenzene (0.1-1%). Product toxicity to daphnia and other aquatic invertebrates: LC50 Ceriodaphnia dubia: 4,063 mg/l Exposure time: 48 hrs Test substance: Product. NOEC Ceriodaphnia dubia: 2,500 mg/l Exposure time: 48 hrs Test substance.
	Corrosion inhibitor 2	Not Classified	No GHS classification provided in Section 2. Product consists of methanol (30-60%); QAC (10-30%); Fatty acid-amine condensate (5-10%); Ethylene Glycol (5-10%); 2-Mercaptoethanol (5-10%);Oxyalkylate (1-5%); Diethanolamine (1-5%); Heavy Aromatic Naphtha (1-5%); and Naphthalene (0.1-1%). Fish and invertebrate L(E)C50s for methanol are >100 mg/L. No fish or invertebrate toxicity data are reported for QAC.
	Oxygen Scavenger 2	Not Assessed	No GHS classification provided in section 2. Substances identified are a proprietary substituted alkylamine (10-30%); ethylene glycol (5-10%); and 2-butoxyethanol (1-5%). Toxicity data identify a 96-h LC50 of >1.908 mg/L for fish exposed to the substituted alkylamine and a 48-h LC50 of 20.352 mg/L for the Daphnid.
	Scale inhibitor 2	Not Assessed	No GHS classification is provided in Section 2. Substances identified are ethylene glycol (10-30%); sodium molybdate (1-5%); and Inorganic salt (0.1-1%). SDS indicates that this product has no known ecotoxicological effects. Fish and invertebrate L(E)C50s for ethylene glycol are >100 mg/L.
	Completion Fluid Additive 1	Not Assessed	No GHS classification is provided in Section 2. Substances identified are calcium bromide (50-60%) and several proprietary compounds (<25%). No toxicity data are provided in Section 12. The section also indicates that the product is not considered harmful to aquatic organisms or to cause long-term adverse effects in the environment.

Table A6. GHS Acute Aquatic Toxicity
Classification of Chemical Products in TCW Category I Effluents

TCW Sample	Product	GHS Acute Aquatic Toxicity Category Classification	Notes
EP57	Defoamer 2	Not Assessed	No GHS classification provided in Section 2. Product consists of kerosene (60-100%); naphthalene (1-5%); and ethylbenzene (0.1-1%). Product toxicity to daphnia and other aquatic invertebrates: LC50 Ceriodaphnia dubia: 4,063 mg/l Exposure time: 48 hrs Test substance: Product. NOEC Ceriodaphnia dubia: 2,500 mg/l Exposure time: 48 hrs Test substance.
	Corrosion inhibitor 2	Not Assessed	No GHS classification provided in Section 2. Product consists of methanol (30-60%); QAC (10-30%); Fatty acid-amine condensate (5-10%); Ethylene Glycol (5-10%); 2-Mercaptoethanol (5-10%);Oxyalkylate (1-5%); Diethanolamine (1-5%); Heavy Aromatic Naphtha (1-5%); and Naphthalene (0.1-1%). Fish and invertebrate L(E)C50s for methanol are >100 mg/L. No fish or invertebrate toxicity data are reported for QAC.
	Oxygen Scavenger 2	Not Assessed	No GHS classification provided in section 2. Substances identified are a proprietary substituted alkylamine (10-30%); ethylene glycol (5-10%); and 2-butoxyethanol (1-5%). Toxicity data identify a 96-h LC50 of >1.908 mg/L for fish exposed to the substituted alkylamine and a 48-h LC50 of 20.352 mg/L for the Daphnid.
	Scale inhibitor 2	Not Assessed	No GHS classification is provided in Section 2. Substances identified are ethylene glycol (10-30%); sodium molybdate (1-5%); and Inorganic salt (0.1-1%). SDS indicates that this product has no known ecotoxicological effects. Fish and invertebrate L(E)C50s for ethylene glycol are >100 mg/L.
	Completion Fluid Additive 1	Not Assessed	No GHS classification is provided in Section 2. Substances identified are calcium bromide (50-60%) and several proprietary compounds (<25%). No toxicity data are provided in Section 12. The section also indicates that the product is not considered harmful to aquatic organisms or to cause long-term adverse effects in the environment.

Table A6. GHS Acute Aquatic Toxicity
Classification of Chemical Products in TCW Category I Effluents

TCW Sample	Product	GHS Acute Aquatic Toxicity Category Classification	Notes
TR84	Defoamer 2	Not Assessed	No GHS classification provided in Section 2. Product consists of kerosene (60-100%); naphthalene (1-5%); and ethylbenzene (0.1-1%). Product toxicity to daphnia and other aquatic invertebrates: LC50 Ceriodaphnia dubia: 4,063 mg/l Exposure time: 48 hrs Test substance: Product. NOEC Ceriodaphnia dubia: 2,500 mg/l Exposure time: 48 hrs Test substance.
	Corrosion inhibitor 2	Not Assessed	No GHS classification provided in Section 2. Product consists of methanol (30-60%); QAC (10-30%); Fatty acid-amine condensate (5-10%); Ethylene Glycol (5-10%); 2-Mercaptoethanol (5-10%);Oxyalkylate (1-5%); Diethanolamine (1-5%); Heavy Aromatic Naphtha (1-5%); and Naphthalene (0.1-1%). Fish and invertebrate L(E)C50s for methanol are >100 mg/L. No fish or invertebrate toxicity data are reported for QAC.
	Oxygen Scavenger 2	Not Assessed	No GHS classification provided in section 2. Substances identified are a proprietary substituted alkylamine (10-30%); ethylene glycol (5-10%); and 2-butoxyethanol (1-5%). Toxicity data identify a 96-h LC50 of >1.908 mg/L for fish exposed to the substituted alkylamine and a 48-h LC50 of 20.352 mg/L for the Daphnid.
	Scale inhibitor 2	Not Assessed	No GHS classification is provided in Section 2. Substances identified are ethylene glycol (10-30%); sodium molybdate (1-5%); and Inorganic salt (0.1-1%). SDS indicates that this product has no known ecotoxicological effects. Fish and invertebrate L(E)C50s for ethylene glycol are >100 mg/L.
	Completion Fluid Additive 1	Not Assessed	No GHS classification is provided in Section 2. Substances identified are calcium bromide (50-60%) and several proprietary compounds (<25%). No toxicity data are provided in Section 12. The section also indicates that the product is not considered harmful to aquatic organisms or to cause long-term adverse effects in the environment.

Table A7. GHS Acute Aquatic Toxicity
Classification of Chemical Products in Category III Effluents

TCW Sample	Chemical Product Code	GHS Acute/Chronic Aquatic Toxicity Category Classification	Notes
JK70	Non-emulsifier 3	Not Assessed	No GHS classification identified in Section 2. Insufficient information provided on composition for proprietary substances (QACs). No ecological data
	Clay Stabilizer 2	Not Assessed	No GHS classification identified in Section 2. Insufficient information provided on composition for proprietary substances (QACs). No ecological data.
	Corrosion Inhibitor 3	Not Assessed	No GHS classification identified in Section 2. Insufficient information provided on composition for proprietary substances (QACs).
	Defoamer 3	Not Assessed	No GHS classification identified in Section 2. Insufficient information provided on composition for proprietary substances (TBP 40-60% w/w). No ecological data.
	Oxygen Scavenger 3	Not Assessed	No GHS classification identified in Section 2. Insufficient information provided on composition for proprietary substances ("proprietary poly-functional organic"). No ecological data.
	Synthetic Mud Casing Scrubber 1	Not Assessed	No GHS classification identified in Section 2. Insufficient information provided on composition for proprietary substances ("surfactant blend"). 96h LC-50 (fish) identified in SDS: >100 mg/L
	Defoamer 4	Not Assessed	No GHS classification identified in Section 2. No information provided on composition for substances. No ecological data.
	Viscosifier 3	Not Assessed	No GHS classification identified in Section 2. No information provided on composition for substances. No ecological data.
	Viscosifier 4	Not Assessed	No GHS classification identified in Section 2. Insufficient information provided on composition. No ecological data.

Table A7. GHS Acute Aquatic Toxicity
Classification of Chemical Products in Category III Effluents

TCW Sample	Product	GHS Acute/Chronic Aquatic Toxicity Category Classification	Notes
JK70	Fluid additive 1	Not Assessed	Identified as Not Classified for environmental hazards in SDS Section 2. Benzenesulfonic acid, C10-16-alkyl derivatives, compounds with 2-Propanamine (10-30% w/w). Environmental hazards identified as "Not classified" in SDS Section 2
	Biocide 5	Not Assessed	No GHS classification identified in Section 2. Substance identified is glutaraldehyde (25% w/w). No ecological data provided in SDS.
NY50	Viscosifier 1	Not Assessed	Identified as Not Classified in SDS Section 2. Contains no hazardous substances in concentrations above cut-off values according to the competent authority. No ecological data.
	Non-emulsifier 1	Acute 2	Identified in SDS Section 2.
YO64	Well Cleaner 1	Not Assessed	No GHS classification provided in Section 2. Manufacturer product toxicity data provided in SDS Section 12 reports product data of: Algae Toxicity EC50 (72h) >10 mg/L (Pseudokirchneriella subcapitata). Acute Crustaceans Toxicity: EC50 (48h) >10 mg/L (Daphnia magna).
	pH Control 3	Not Assessed	No GHS classification for aquatic toxicity identified in Section 2 or for individual substances in Section 3. Product contains sodium hydroxide (10-30%); no other substances identified. 24/48/96-h LC50s for fish range from 125-189 mg/L; 48-h EC50 for Ceriodaphnia sp. Is 40.4 mg/L.
	Non-emulsifier 1	Acute 2	Identified in SDS Section 2.
	Viscosifier 2	Not Assessed	No GHS classification provided in Section 2. No GHS classification for individual substances (dipropylene glycol monomethyl ether; 30-60%) provided in Section 3.
	Biocide 4	Acute 3	Identified in SDS Section 2.

Table A7. GHS Acute Aquatic Toxicity
Classification of Chemical Products in Category III Effluents

TCW Sample	Product	GHS Acute/Chronic Aquatic Toxicity Category Classification	Notes
FP89	Surfactant 1	Not Assessed	No GHS classification provided in Section 2. Amphoteric surfactant. Only 1 substance identifed (cocamidopropyl betaine 10 - 20%). No ecological data provided.
	Biocide 3	Acute 1	Identified in SDS Section 2.
	Linear gel 1	Not Assessed	No GHS classification provided in Section 2. SDS indicates that the product contains no hazardous substances. No information on composition. No ecological information
	Breaker 2	Not Assessed	No GHS classification provided in Section 2. Limited composition information (hemicellulase enzyme; 0.1 - 1%). No ecological data provided.
	Gellant 2	Not Assessed	No GHS classification provided in Section 2. Product consists of glycol ether (60 - 65%) and guar gum (30 - 35%). No ecological data provided.
	Non-emulsifier 2	Not Assessed	No GHS classification provided in Section 2. Substances identified include a cationic polymer in solution (1-5%); and ethoxylated alcohol (1-5%). No CAS Nos. provided.
	pH Control 2	Not Assessed	No GHS classification provided in Section 2. Percentage of the mixture consisting of ingredient(s) of unknown hazards to the aquatic environment: 2%. Product contains Potassium carbonate (40 - 50%); this is the only substance identified.
	Biocide 2	Acute 1	Identified in SDS Section 2.
	Clay Stabilizer 1	Not Assessed	No GHS classification provided in Section 2. Ammonium chloride (1-5%) is the only substance identified. No ecotoxicological information provided.

Table A7. GHS Acute Aquatic Toxicity
Classification of Chemical Products in Category III Effluents

TCW Sample	Product	GHS Acute/Chronic Aquatic Toxicity Category Classification	Notes
FP89	Acid 6	Not Assessed	No GHS classification identified in Section 2. Substances identified are ammonium bifluoride (1-5%); acetic anhydride (1-5%); acetic acid (1-5%); hydrochloric acid (5-10%); hydrofluoric acid (1-5%). No ecological information provided.
	Solvent 1	Not Assessed	No GHS classification identified in Section 2. Substances identified are xylene (70-80%); acetic acid (10-20%); 2-Butoxyethanol (10-20%). LC50 data identified in Section 12 for xylene identify a 96h LC50 of 2.6 mg/L for fish and a 48-h LC50 of >3.4 mg/L for Daphnia.
GQ67	Surfactant 2	Not Assessed	No GHS classification provided in Section 2. Substances identified are alcohols (C9-11 ethoxylated) (10%) and proprietary organic alcohol (10-30%). No toxicity data are available.
YU91	Clay Stabilizer 1	Not Assessed	No GHS classification provided in Section 2. Ammonium chloride (1-5%) is the only substance identified. No ecotoxicological information provided.
	pH Control 1	Not Assessed	No GHS classification is provided in Section 2. Substances identified are sodium carbonate (60-100%).
	Acid 6	Not Assessed	No GHS classification identified in Section 2. Substances identified are ammonium bifluoride (1-5%); acetic anhydride (1-5%); acetic acid (1-5%); hydrochloric acid (5-10%); hydrofluoric acid (1-5%). No ecological information provided.
	Linear Gel 2	Not Assessed	No GHS classification provided in Section 2. Substance identified is ulexite (0.1-1%).
	Fluid Additive 3	Not Assessed	SDS Section 2 indicates that the product is not classified. The SDS also indicates that the product contains no hazardous substances in concentrations above cut-off values according to the competent authority
LX98	Oxygen Scavenger 1	Acute 3	Identified in SDS Section 2.
IS88	Oxygen Scavenger 1	Acute 3	Identified in SDS Section 2.

Table A7. GHS Acute Aquatic Toxicity
Classification of Chemical Products in Category III Effluents

TCW Sample	Product	GHS Acute/Chronic Aquatic Toxicity Category Classification	Notes
IS88	pH Control 3	Not Assessed	No GHS classification for aquatic toxicity identified in Section 2 or for individual substances in Section 3. Product contains sodium hydroxide (10-30%); no other substances identified. 24/48/96-h LC50s for fish range from 125-189 mg/L; 48-h EC50 for Ceriodaphnia sp. is 40.4 mg/L.
	Biocide 4	Acute 3	Identified in SDS Section 2.
RU72	No SDSs provided	--	--
BT52	No SDSs provided	--	--
SH87	Acid 3	Not Assessed	No GHS classification is provided in Section 2. Substances identified are acetic acid (5-10%). This is an organic acid (aqueous solution).
	Acid 7	Not Assessed	Section 2 indicates that the product is not classified. Substances identified are ammonium chloride (3-6%).
	Acid 4	Not Assessed	No GHS classification is provided in Section 2. The substance identified is hydrogen chloride (5-10%).
	Acid 1	Not Assessed	No GHS classification is provided in Section 2. Substances identified are hydrogen chloride (5-10%) and Hydrofluoric acid (1-5%).
	Biocide 2	Acute 1	Identified in SDS Section 2.

Table A7. GHS Acute Aquatic Toxicity
Classification of Chemical Products in Category III Effluents

TCW Sample	Product	GHS Acute/Chronic Aquatic Toxicity Category Classification	Notes
SH87	pH Control 2	Not Assessed	No GHS classification provided in Section 2. Substance identified is potassium carbonate. Percentage of the mixture consisting of ingredient(s) of unknown hazards to the aquatic environment: 2%.
	Proppant 1	Not Assessed	This product is proppant. No applicable toxicity data.
	Proppant 2	Not Assessed	This product is proppant. No applicable toxicity data.
	Corrosion Inhibitor 1	Acute 2	Identified in SDS Section 2.
	Corrosion Inhibitor 5	Acute 1	Identified in SDS Section 2.
	Breaker 2	Not Assessed	No GHS classification provided in Section 2. Limited composition information (hemicellulase enzyme; 0.1 - 1%). No ecological data provided.
	Iron Control 1	Not Assessed	No GHS classification provided in Section 2. The single substance identified is citric acid (40-50%). An Acute 48-LC50 of 160 mg/l was reported in SDS Section 12 for the marine crustacean (adult *Carcinus maenas*) exposed to citric acid.
	Surfactant 1	Not Assessed	No GHS classification provided in Section 2. Amphoteric surfactant. Only 1 substance identifed (cocamidopropyl betaine 10 - 20%). SDS Section 12 indicates that the product is harmful to aquatic organisms, may cause long-term adverse effects in the aquatic environment. No ecological data provided.
	Breaker 3	Not Assessed	No GHS classification provided in Section 2. The only substance identified is sodium chlorite (5-10%). A 48-h EC50 for sodium chlorite reported for Daphnia is 0.025 mg/L. An acute 96-h LC50 reported for fish is 0.08 mg/L. Percentage of the mixture consisting of ingredient(s) of unknown hazards to the aquatic environment: 12.5%
	Breaker 4	Not Assessed	No GHS classification provided in Section 2. Substance identified is ammonium persulphate (90 - 100%). The minimum acute 96-h LC50 identified in SDS Section 12 is 76.3 mg/l reported for freshwater fish. SDS Section 12 indicates no known significant effects or critical hazards.
	Gellant 2	Not Assessed	No GHS classification provided in Section 2. Product consists of glycol ether (60 - 65%) and guar gum (30 - 35%). No ecological data provided. SDS Section 12 indicates no known significant effects or critical hazards.

Table A7. GHS Acute Aquatic Toxicity
Classification of Chemical Products in Category III Effluents

TCW Sample	Product	GHS Acute/Chronic Aquatic Toxicity Category Classification	Notes
SH87	Gellant 1	Not Assessed	No GHS classification provided in Section 2. Consists of alkoxylated alcohol (60-70%). Percentage of the mixture consisting of ingredient(s) of unknown hazards to the aquatic environment: 62.5%. SDS Section 12 indicates "no known significant effects or critical hazards".
	Corrosion Inhibitor 4	Not Assessed	No GHS classification provided in Section 2. Substances identified are potassium iodide (90 - 100%). SDS Section 12 identifies an acute 96-h LC50 for potassium iodide of 896 mg/L for the freshwater fish *Oncorhynchus mykiss* . Acute aquatic toxicity is not expected.
	Non-emulsifier 2	Not Assessed	No GHS classification provided in Section 2. The only substances identified in the SDS are a cationic polymer in solution (1-5%); and ethoxylated alcohol (1-5%). No CAS Nos. provided. The minimum acute 96-h LC50 of 0.6 mg/l was identified in SDS Section 12 for a freshwater fish. SDS Section 12 also indicates "no known significant effects or critical hazards".
	Solvent 1	Not Assessed	No GHS classification identified in Section 2. Substances identified are xylene (70-80%); acetic acid (10-20%); 2-Butoxyethanol (10-20%). LC50 data identified in Section 12 for xylene identify a 96h LC50 of 2.6 mg/L for fish and a 48-h LC50 of >3.4 mg/L for Daphnia.
	Surfactant 3	Not Assessed	Product consists of 100% poly(oxy-1,2-ethanediyl), a-undecyl-w-hydroxy (CAS No. 34398-01-1). SDS Section 12 indicates no data on product is available, but a 96-h LC50 reported for *Pimephales promelas* (fathead minnow) is 1 - 10 mg/l.
	Crosslinker 2	Not Assessed	No GHS classification identified in Section 2. Percentage of the mixture consisting of ingredient(s) of unknown hazards to the aquatic environment: 6.2%. Substances >25% are glyoxal (20-30%). The glyoxal 96-h LC50 for Pimephales promelas is 215 mg/L. Other substances are sodium tetraborate (10-20%) and sodium hydroxide (1-5%). An Acute 48-h EC50 of 1,645 mg sodium tetraborate/L was reported for the freshwater crustacean (Cypris subglobosa) exposed. The minimum acute 48-h EC50 of 40.38 mg sodium hydroxide/L was reported for the freshwater crustacean (neonate *Ceriodaphnia dubia*).
RC74	Crosslinker 1	Not Assessed	No GHS classification is provided in Section 2. Substances identified are borate salts (30-60%) and dipropylene glycol monomethyl ether (30-60%). Borate salts: Acute 96-h LC50 for fish are >100 mg/L; 48-h EC50 for invertebrates are >100 mg/L. Dipropylene glycol monomethyl ether: a NOEC of 0.5 mg/L was reported for Daphnia magna; no fish data are available.

Table A7. GHS Acute Aquatic Toxicity
Classification of Chemical Products in Category III Effluents

TCW Sample	Product	GHS Acute/Chronic Aquatic Toxicity Category Classification	Notes
RC74	Stabilizer 1	Not Assessed	This product is identified in Section 2 as not classified. No composition or toxicity data are available.
	Fluid Additive 2	Not Assessed	No GHS classification identified in Section 2. Substances identified are borate salts (0.1-1%) and diesel (0.1-1%).Borate salts: Acute 96-h LC50 for fish are >100 mg/L; 48-h EC50 for invertebrates are >100 mg/L. Diesel: LC50 for fish = 35 mg/L and an LL50 (96h) of 21 mg/L (*Oncorhynchus mykiss*); 48-h EL50 for Daphnia magna is 210 mg/L.
	Proppant 3	Not Assessed	This product is proppant. No applicable toxicity data.
	Scale Inhibitor 1	Not Assessed	No GHS classification is identified in Section 2. Substances identified are ethylene glycol (10-30%) and methanol (0.1-1%). Acute LC50s for fish and invertebrates exposed to ethylene glycol are >100 mg/L. Acute toxicity is not expected. Also, SDS Section 12 indicates that this product has no known ecotoxicological effects
	Oil Tracer 1	No Acute Classification	Chronic toxicity identified in SDS Section 2 only; an acute classification was not identified.
	Defoamer 5	Not Assessed	No GHS classification was identified in Section 2. There was no information on composition or aquatic toxicity. SDS Section 12 indicates that the environmental impact of this product has not been fully investigated.
	Diagnostic Additive 1	Not Assessed	No GHS classification was identified in Section 2. There was no information on composition or aquatic toxicity.
	Breaker 1	Acute 2	Identified in SDS Section 2.
	Fluid Additive 3	Not Assessed	No GHS classification was identified in Section 2. There was no information on composition or aquatic toxicity.
OD76	Crosslinker 1	Not Assessed	No GHS classification is provided in Section 2. Substances identified are borate salts (30-60%) and dipropylene glycol monomethyl ether (30-60%). Borate salts: Acute 96-h LC50 for fish are >100 mg/L; 48-h EC50 for invertebrates are >100 mg/L. Dipropylene glycol monomethyl ether: a NOEC of 0.5 mg/L was reported for Daphnia magna; no fish data are available.
	Stabilizer 1	Not Assessed	This product is identified in Section 2 as not classified. No composition or toxicity data are available.
	Fluid Additive 2	Not Assessed	No GHS classification identified in Section 2. Substances identified are borate salts (0.1-1%) and diesel (0.1-1%).Borate salts: Acute 96-h LC50 for fish are >100 mg/L; 48-h EC50 for invertebrates are >100 mg/L. Diesel: LC50 for fish = 35 mg/L and an LL50 (96h) of 21 mg/L (Oncorhynchus mykiss); 48-h EL50 for Daphnia magna is 210 mg/L.
	Proppant 3	Not Assessed	This product is proppant. No applicable toxicity data.

Table A7. GHS Acute Aquatic Toxicity
Classification of Chemical Products in Category III Effluents

TCW Sample	Product	GHS Acute/Chronic Aquatic Toxicity Category Classification	Notes
OD76	Scale Inhibitor 1	Not Assessed	No GHS classification is identified in Section 2. Substances identified are ethylene glycol (10-30%) and methanol (0.1-1%). Acute LC50s for fish and invertebrates exposed to ethylene glycol are >100 mg/L. Acute toxicity is not expected. Also, SDS Section 12 indicates that this product has no known ecotoxicological effects
	Oil Tracer 1	No Acute Classification	Chronic toxicity Identified in SDS Section 2 only; an acute classification was not identified.
	Defoamer 5	Not Assessed	No GHS classification was identified in Section 2. There was no information on composition or aquatic toxicity. SDS Section 12 indicates that the environmental impact of this product has not been fully investigated.
	Diagnostic Additive 1	Not Assessed	No GHS classification was identified in Section 2. There was no information on composition or aquatic toxicity.
	Breaker 1	Acute 2	Identified in SDS Section 2.
	Fluid Additive 3	Not Assessed	No GHS classification was identified in Section 2. There was no information on composition or aquatic toxicity.
TF74	Crosslinker 1	Not Assessed	No GHS classification is provided in Section 2. Substances identified are borate salts (30-60%) and dipropylene glycol monomethyl ether (30-60%). Borate salts: Acute 96-h LC50 for fish are >100 mg/L; 48-h EC50 for invertebrates are >100 mg/L. Dipropylene glycol monomethyl ether: a NOEC of 0.5 mg/L was reported for Daphnia magna; no fish data are available.
	Stabilizer 1	Not Assessed	This product is identified in Section 2 as not classified. No composition or toxicity data are available.
	Fluid Additive 2	Not Assessed	No GHS classification identified in Section 2. Substances identified are borate salts (0.1-1%) and diesel (0.1-1%).Borate salts: Acute 96-h LC50 for fish are >100 mg/L; 48-h EC50 for invertebrates are >100 mg/L. Diesel: LC50 for fish = 35 mg/L and an LL50 (96h) of 21 mg/L (Oncorhynchus mykiss); 48-h EL50 for Daphnia magna is 210 mg/L.
	Proppant 3	Not Assessed	This product is proppant. No applicable toxicity data.
	Scale Inhibitor 1	Not Assessed	No GHS classification is identified in Section 2. Substances identified are ethylene glycol (10-30%) and methanol (0.1-1%). Acute LC50s for fish and invertebrates exposed to ethylene glycol are >100 mg/L. Acute toxicity is not expected. Also, SDS Section 12 indicates that this product has no known ecotoxicological effects

Table A7. GHS Acute Aquatic Toxicity
Classification of Chemical Products in Category III Effluents

TCW Sample	Product	GHS Acute/Chronic Aquatic Toxicity Category Classification	Notes
TF74	Oil Tracer 1	No Acute Classification	Chronic toxicity Identified in SDS Section 2; an acute classification was not identified.
	Defoamer 5	Not Assessed	No GHS classification was identified in Section 2. There was no information on composition or aquatic toxicity. SDS Section 12 indicates that the environmental impact of this product has not been fully investigated.
	Diagnostic Additive 1	Not Assessed	No GHS classification was identified in Section 2. There was no information on composition or aquatic toxicity.
	Breaker 1	Acute 2	Identified in SDS Section 2.
	Fluid Additive 3	Not Assessed	No GHS classification was identified in Section 2. There was no information on composition or aquatic toxicity.

Constituent	Units	Priority Pollutant?	Published/Promulgated USEPA ESVs		*Americamysis bahia* -specific 48-h LC50s for Ions			*Menidia beryllina* -specific 48-h LC50s for Ions		
			Acute ESV (mg/L)	Acute ESV Source	Ion Deficiency	Ion Excess	Source	Ion Deficiency	Ion Excess	Source
Critical Effluent Dilution	%	--	--	--	--	--	--	--	--	--
Date	--	--	--	--	--	--	--	--	--	--
Water Quality Parameters (Total)										
Hardness (as CaCO3)	mg/L	No	--	--	--	--	--	--	--	--
Alkalinity, Total (As CaCO3)	mg/L	No	No published / promulgated Acute Saltwater ESV	--	--	--	--	--	--	--
HCO_3^- (Estimated as 1.22 * Total Alk.)	mg/L	No	No published / promulgated Acute Saltwater ESV	--	--	1090	Pillard et al., 2000	--	670	Pillard et al., 2000
Total Suspended Solids (Residue, Non-Filterable)	mg/L	No	--	--	--	--	--	--	--	--
Nitrogen, Ammonia (As N)[1]	mg/L	No	5	USEPA. 1989. NRALC Ammonia (Saltwater): Acute CMC: pH = 8; Temp. 25 Deg. C; and salinity = 30 ppt[1]	--	--	--	--	--	--
Chemical Oxygen Demand	mg/L	No	--		--	--	--	--	--	--
Organic Carbon, Total	mg/L	No	--	--	--	--	--	--	--	--
Sulfide	mg/L	No	No published / promulgated Acute Saltwater ESV	--	--	--	--	--	--	--
Specific Gravity										
Water Quality Parameters (Dissolved)										
Total Dissolved Solids (Residue, Filterable)	mg/L	No	--	--	--	--	--	--	--	--
Dissolved Organic Carbon	mg/L	No	--	--	--	--	--	--	--	--
Metals (Total)										
As	mg/L	Yes	0.069	USEPA NRALC: Saltwater	--	--	--	--	--	--
Ba	mg/L	No	No published / promulgated Acute Saltwater ESV	--	--	--	--	--	--	--
Cd	mg/L	Yes	0.0402	USEPA Region IV ESV: Saltwater	--	--	--	--	--	--
Ca	mg/L	No	No published / promulgated Acute Saltwater ESV	--	100	1100	Pillard et al., 2000	10	4610	Pillard et al., 2000
Cr	mg/L	Yes	0.515	USEPA Region IV ESV: Saltwater	--	--	--	--	--	--
Cu	mg/L	Yes	0.0056	USEPA Region IV ESV: Saltwater	--	--	--	--	--	--
Pb	mg/L	Yes	0.22	USEPA Region IV ESV: Saltwater	--	--	--	--	--	--
Mg	mg/L	No	No published / promulgated Acute Saltwater ESV	--	--	2650	Pillard et al., 2000	--	2800	Pillard et al., 2000
Hg	mg/L	Yes	0.0018	USEPA NRALC: Saltwater	--	--	--	--	--	--
Ni	mg/L	Yes	0.075	USEPA Region IV ESV: Saltwater	--	--	--	--	--	--

Table A8. Acute Aquatic Life Ecological Screening Values

Constituent	Units	Priority Pollutant?	Published/Promulgated USEPA ESVs		*Americamysis bahia*-specific 48-h LC50s for Ions			*Menidia beryllina*-specific 48-h LC50s for Ions		
			Acute ESV (mg/L)	Acute ESV Source	Ion Deficiency	Ion Excess	Source	Ion Deficiency	Ion Excess	Source
K	mg/L	No	No published / promulgated Acute Saltwater ESV	--	115	790	Pillard et al., 2000	--	1100	Pillard et al., 2000
Se	mg/L	Yes	0.29	USEPA NRALC: Saltwater	--	--	--	--	--	--
Na	mg/L	No	No published / promulgated Acute Saltwater ESV	--	--	--	--	--	--	--
Tl	mg/L	Yes	0.71	USEPA Region IV ESV: Saltwater	--	--	--	--	--	--
Zn	mg/L	Yes	0.092	USEPA Region IV ESV: Saltwater	--	--	--	--	--	--
Metals (Dissolved)										
As	mg/L	Yes	0.069	USEPA NRALC: Saltwater	--	--	--	--	--	--
Ba	mg/L	No	No published / promulgated Acute Saltwater ESV	--	--	--	--	--	--	--
Cd	mg/L	Yes	0.033	USEPA NRALC: Saltwater	--	--	--	--	--	--
Ca	mg/L	No	No published / promulgated Acute Saltwater ESV	--	100	1100	Pillard et al., 2000	--	4610	Pillard et al., 2000
Cr	mg/L	Yes	0.515	USEPA Region IV ESV: Saltwater	--	--	--	--	--	--
Cu	mg/L	Yes	0.0048	USEPA NRALC: Saltwater	--	--	--	--	--	--
Pb	mg/L	Yes	0.14	USEPA NRALC: Saltwater	--	--	--	--	--	--
Mg	mg/L	No	No published / promulgated Acute Saltwater ESV	--	--	2650	Pillard et al., 2000	--	2800	Pillard et al., 2000
Hg	mg/L	Yes	0.0018	USEPA NRALC: Saltwater	--	--	--	--	--	--
Ni	mg/L	Yes	0.074	USEPA NRALC: Saltwater	--	--	--	--	--	--
K	mg/L	No	--	--	115	790	Pillard et al., 2000	--	1100	Pillard et al., 2000
Se	mg/L	Yes	0.29	USEPA NRALC: Saltwater	--	--	--	--	--	--
Na	mg/L	No	No published / promulgated Acute Saltwater ESV	--	--	--	--	--	--	--
Tl	mg/L	Yes	0.71	USEPA Region IV ESV: Saltwater	--	--	--	--	--	--
Zn	mg/L	Yes	0.09	USEPA NRALC: Saltwater	--	--	--	--	--	--
Inorganic Anions (Total)										
Br	mg/L	No	No published / promulgated Acute Saltwater ESV	--	--	7990	Pillard et al., 2000	--	18300	Pillard et al., 2000

Table A8. Acute Aquatic Life Ecological Screening Values

Constituent	Units	Priority Pollutant?	Published/Promulgated USEPA ESVs		*Americamysis bahia* -specific 48-h LC50s for Ions			*Menidia beryllina* -specific 48-h LC50s for Ions		
			Acute ESV (mg/L)	Acute ESV Source	Ion Deficiency	Ion Excess	Source	Ion Deficiency	Ion Excess	Source
Inorganic Anions (Total)										
Cl	mg/L	No	No published / promulgated Acute Saltwater ESV	--	--	--	--	--	--	--
SO_4^{2-}	mg/L	No	No published / promulgated Acute Saltwater ESV	--	--	16710	Pillard et al., 2000	--	26710	Pillard et al., 2000
Polycyclic Aromatic Hydrocarbons (PAHs)										
Acenaphthene	mg/L	Yes	0.32	USEPA Region IV ESV: Saltwater	--	--	--	--	--	--
Acenaphthylene	mg/L	Yes	0.291	USEPA Region IV ESV: Saltwater	--	--	--	--	--	--
Anthracene	mg/L	Yes	0.0018	USEPA Region IV ESV: Saltwater	--	--	--	--	--	--
Benzo(a)anthracene	mg/L	Yes	0.0046	USEPA Region IV ESV: Saltwater	--	--	--	--	--	--
Benzo(a)pyrene	mg/L	Yes	0.00064	USEPA Region IV ESV: Saltwater	--	--	--	--	--	--
Benzo(b)fluoranthene	mg/L	Yes	0.0014	USEPA Region IV ESV: Saltwater	--	--	--	--	--	--
Benzo(g,h,i)perylene	mg/L	Yes	0.00019	USEPA Region IV ESV: Saltwater	--	--	--	--	--	--
Benzo(k)fluoranthene	mg/L	Yes	0.0013	USEPA Region IV ESV: Saltwater	--	--	--	--	--	--
Chrysene	mg/L	Yes	0.0042	USEPA Region IV ESV: Saltwater	--	--	--	--	--	--
Dibenzo(a,h)anthracene	mg/L	Yes	0.00028	USEPA Region IV ESV: Saltwater	--	--	--	--	--	--
Fluoranthene	mg/L	Yes	0.0034	USEPA Region IV ESV: Saltwater	--	--	--	--	--	--
Fluorene	mg/L	Yes	0.082	USEPA Region IV ESV: Saltwater	--	--	--	--	--	--
Indeno(1,2,3-cd)pyrene	mg/L	Yes	0.00027	USEPA Region IV ESV: Saltwater	--	--	--	--	--	--
Naphthalene	mg/L	Yes	0.78	USEPA Region IV ESV: Saltwater	--	--	--	--	--	--
Phenanthrene	mg/L	Yes	0.0077	USEPA Region IV ESV: Saltwater	--	--	--	--	--	--
Pyrene	mg/L	Yes	0.00045	USEPA Region IV ESV: Saltwater	--	--	--	--	--	--

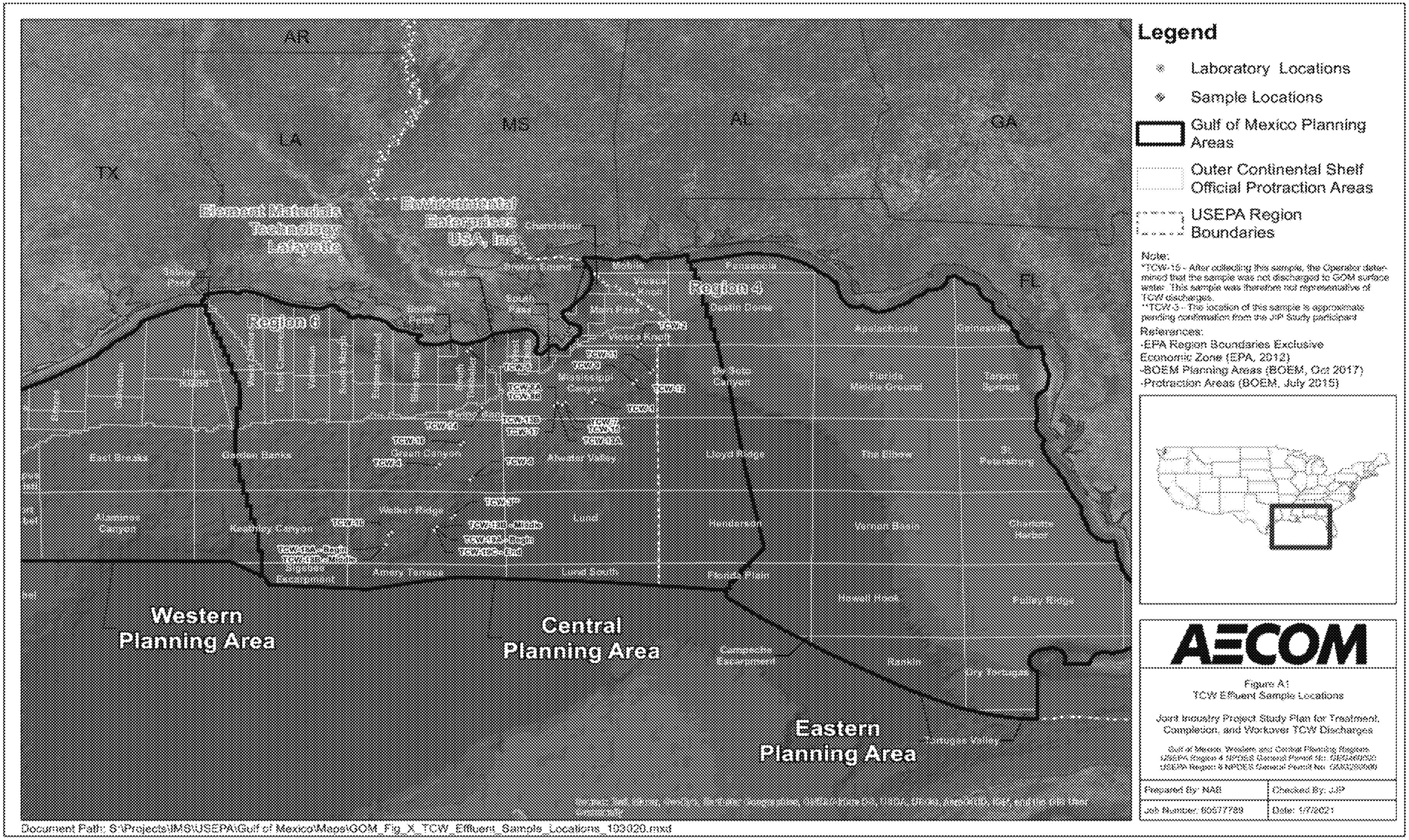
Notes:
CMC; criteria maximum concentration
ESV; ecological screening value
h; hour
LC50; 50% lethal concentration
NRALC; National Recommended Aquatic Life Criteria
USEPA; Unites States Environmental Protection Agency
mg/L; milligrams per liter

[1].

Source	pH (S.U.)	Salinity (ppt)	Temp. (°C)
Maximum of Laboratory Control	7.9	26	26
Closest Values in USEPA 1989	8	30	25

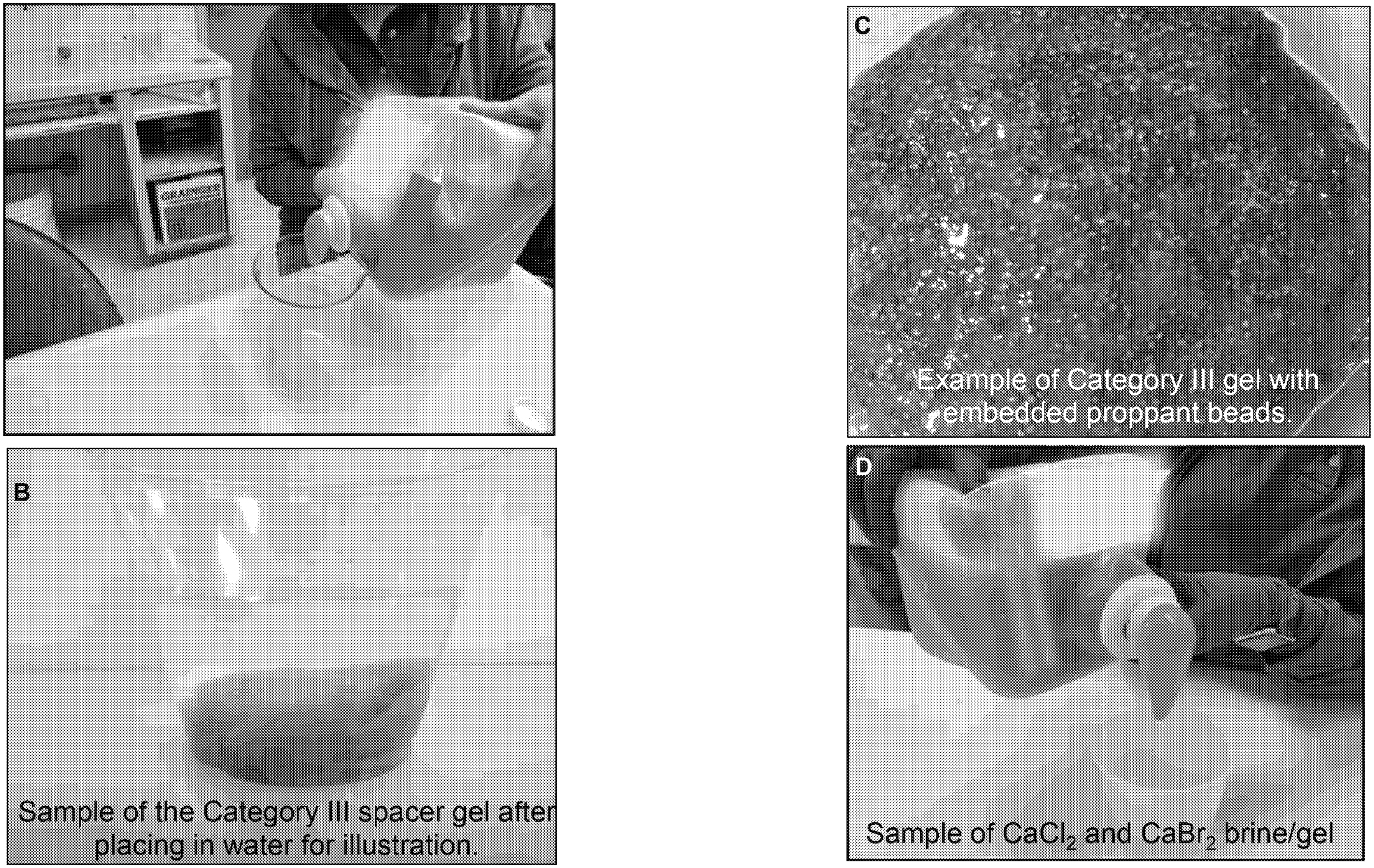
Appendix Figures

Figure A1. TCW Effluent Sample Locations



ED_005567_00000011-00106

Figure A2. Photographs of TCW Category III Gels

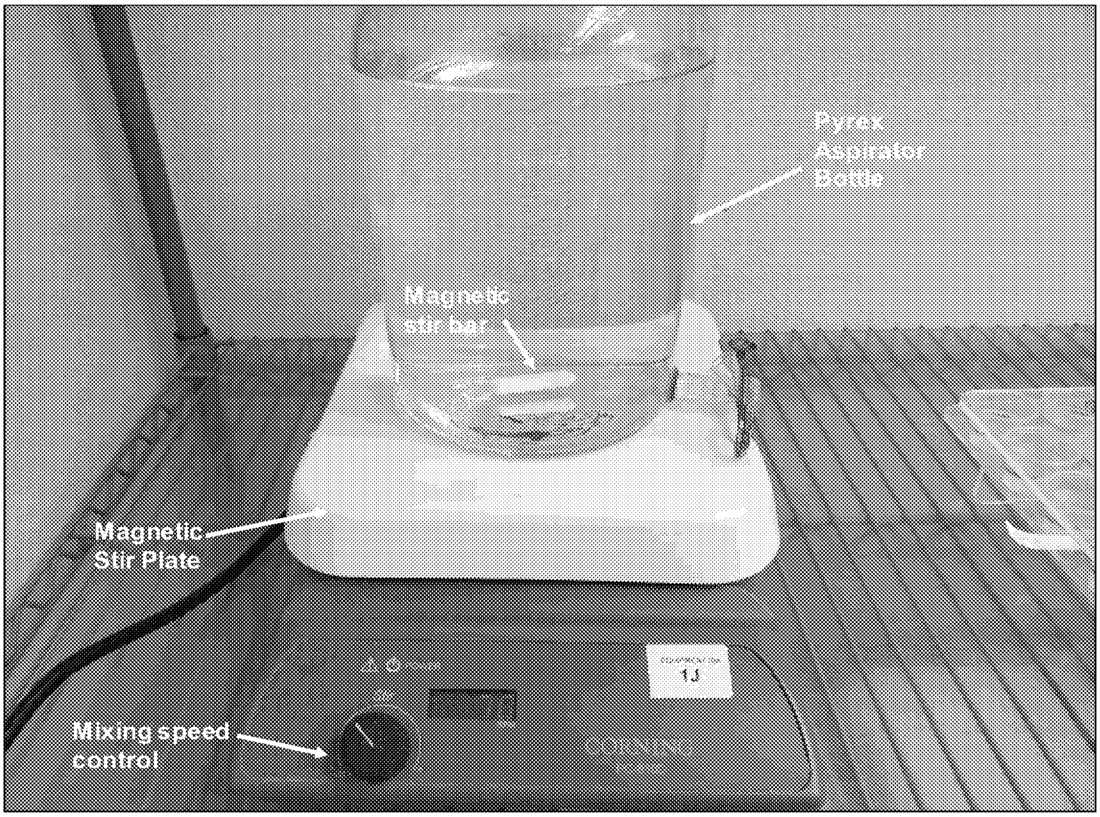


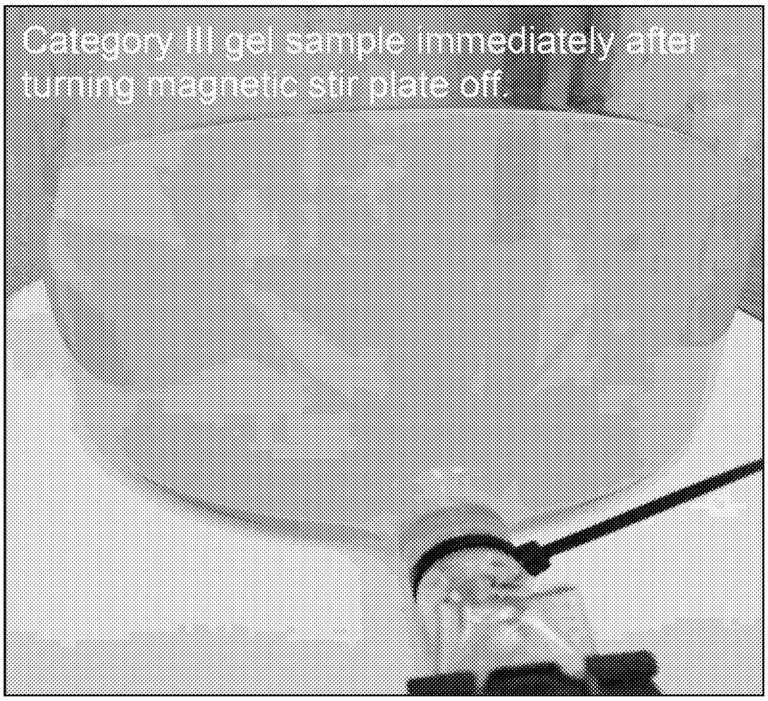
Sample of the Category III spacer gel after placing in water for illustration.

Example of Category III gel with embedded proppant beads.

Sample of $CaCl_2$ and $CaBr_2$ brine/gel

Figure A3. Mixing Apparatus for TCW Category III Gel Samples





Appendices

Appendix A
JIP Study Participant
Survey Questionnaire Form

Appendix A. JIP Study Participant Survey Questionnaire Form

Job Number:
Historical, Existing or Planned?
Date or Anticipated Start Date:
SECTION 1. General Information
1. Contact Name:
2. Telephone Number:
3. Email:
4. Lease:
5. Field:
6. Operator Field:
7. Area:
8. Block:
9. API Well Number:
10. Latitude:
11. Longitude:
12. Permitted Feature Number (if available)
13. Water Column Depth:
SECTION 2. Treatment Completion and Workover (TCW) Fluids
1. What type of well treatment or workover operation is conducted? Please provide a brief description:
2. What types of TCW fluids are used?
a. Category I
b. Category II
c. Category III
d. Category IV
e. Other:
3. Are there jobs where one, or a combination of TCW fluid categories are discharged to surface waters? If yes, proceed to Section 3.
SECTION 3. Discharge of TCW Wastewaters to Surface Water
1. Are TCW wastewaters commingled and discharged as part of produced water?
2. Are TCW wastewaters discharged directly to surface water without treatment or storage in a tank?
a. If yes, is a NPDES-designated discharge point used, e.g., pipe?
b. What is the pipe diameter (inches)?
3. Are TCW wastewaters discharged to a tank on the Facility and then discharged overboard?
a. If yes, is a NPDES-designated discharge point used, e.g., pipe?
b. What is the pipe diameter (inches)?
4. Are TCW wastewaters discharged via a hose off the tank?
a. If yes, what is the hose diameter (inches)?
5. Are the TCW wastewaters discharged above the ocean surface?
a. If yes, at what height above the water column does the discharge occur?
b. If no, at what water column depth does the discharge occur?
6. Typically, how often are TCW wastewaters discharged, e.g., once a week, quarterly?
7. Typically, what is the duration of the discharge (minutes/hours)?
8. Are TCW wastewaters discharged back to the Facility and passed through a filtration system before discharging overboard?
a. Do you use a designated discharge point such as a pipe, if so, what is the diameter (in.)?
b. Do you use a hose off of the Filtration system, if so what is the diameter (in.)?
c. Are wastewaters discharged via any other structure, e.g., diffuser? If yes, please describe:
9. Is any other treatment of TCW wastewaters conducted? If yes, please describe:

SECTION 4. Discharge of Other Wastewaters (Zinc Bromide; Acid Jobs; Chemical Additives) to Surface Water
1. Are zinc bromide wastewaters sent onshore for disposal?
a. If no, how are zinc bromide wastewaters disposed?
b. Other:
2. **Applicable to TCW jobs only**: Are acid jobs conducted? If yes, how are acidic wastewaters treated?
a. Do you send onshore for disposal?
b. Do you discharge acid job wastewaters directly overboard without treatment?
c. Do you neutralize the pH and then discharge overboard?
d. Other:
3. **Applicable to TCW jobs only**: Is there the potential for corrosion inhibitors, deemulsifiers, surfactants, defoamers, or biocides to be comingled with TCW wastewaters? If yes, please identify the type:
a. Corrosion inhibitor:
b. Deemulsifier:
c. Surfactants:
d. Defoamers:
e. Biocides:
f. Other:

Appendix B
Raw Output for Latin Hypercube Sampling Evaluation

Appendix B.1. Characteristics of TCW Discharges Selected by LHS

Area		Mississippi Canyon	Green Canyon	Mississippi Canyon	Mississippi Canyon
Block		392	640	807	809
Water Column Depth (ft.)		7210	4250	2940	3650
Anticipated TCW Fluid Category[1]	I	1	1	1	1
	II	0	1	1	1
	III	0	1	1	1
	IV	0	1	0	0
Treatment Type	No Treatment/Tank Storage	0	0	0	0
	Tank Storage	1	1	1	0
	Filtration	1	0	0	0
	Other Treatment	0	0	0	0
Type of Chemical Additives	Corrosion Inhibitor	0	0	1	0
	De-emulsifier	1	1	1	0
	Surfactants	1	1	1	0
	Defoamers	1	0	1	0
	Biocides	1	1	1	0

Notes:
A "0" indicates the variable was absent; a "1" indicates the variable was present.
[1] Category II and IV effluents were not discharged to Gulf of Mexico surface waters during the Year 1 study.

Appendix B.1. Characteristics of TCW Discharges Selected by LHS

Area		Mississippi Canyon	Mississippi Canyon	Ewing Bank	Mississippi Canyon
Block		437	807	873	807
Water Column Depth (ft.)		7344	2940	773	2940
Anticipated TCW Fluid Category[1]	I	1	1	1	1
	II	0	1	1	1
	III	0	1	1	1
	IV	0	0	0	0
Treatment Type	No Treatment/Tank Storage	0	0	0	0
	Tank Storage	1	1	1	1
	Filtration	0	0	0	0
	Other Treatment	1	0	0	0
Type of Chemical Additives	Corrosion Inhibitor	0	1	0	1
	De-emulsifier	1	1	0	1
	Surfactants	1	1	0	1
	Defoamers	1	1	0	1
	Biocides	1	1	0	1

Notes:
A "0" indicates the variable was absent; a "1" indicates the variable was present.
[1] Category II and IV effluents were not discharged to Gulf of Mexico surface waters during the Year 1 study.

Appendix B.1. Characteristics of TCW Discharges Selected by LHS

Area		Walker Ridge	Walker Ridge	Green Canyon	Mississippi Canyon
Block		29	508	338	392
Water Column Depth (ft.)		5190	9558	3330	7210
Anticipated TCW Fluid Category[1]	I	1	1	1	1
	II	1	1	1	0
	III	1	1	1	0
	IV	1	0	1	0
Treatment Type	No Treatment/Tank Storage	0	0	0	0
	Tank Storage	1	1	0	1
	Filtration	0	0	0	1
	Other Treatment	0	0	0	0
Type of Chemical Additives	Corrosion Inhibitor	0	1	1	0
	De-emulsifier	1	1	1	1
	Surfactants	1	1	1	1
	Defoamers	0	1	1	1
	Biocides	1	1	1	1

Notes:
A "0" indicates the variable was absent; a "1" indicates the variable was present.
[1] Category II and IV effluents were not discharged to Gulf of Mexico surface waters during the Year 1 study.

Appendix B.1. Characteristics of TCW Discharges Selected by LHS

Area		Mississippi Canyon	Green Canyon	Green Canyon	Mississippi Canyon
Block		809	824	825	807
Water Column Depth (ft.)		3600	4976	4976	3030
Anticipated TCW Fluid Category[1]	I	1	1	1	1
	II	1	0	0	1
	III	1	1	1	1
	IV	0	1	1	0
Treatment Type	No Treatment/Tank Storage	0	1	1	0
	Tank Storage	0	0	0	1
	Filtration	0	0	0	0
	Other Treatment	0	0	0	0
Type of Chemical Additives	Corrosion Inhibitor	0	1	1	1
	De-emulsifier	0	0	0	1
	Surfactants	0	1	1	1
	Defoamers	0	1	1	1
	Biocides	0	1	1	1

Notes:
A "0" indicates the variable was absent; a "1" indicates the variable was present.
[1] Category II and IV effluents were not discharged to Gulf of Mexico surface waters during the Year 1 study.

Appendix B.1. Characteristics of TCW Discharges Selected by LHS

Area		Mississippi Canyon	Mississippi Canyon	Green Canyon	Mississippi Canyon
Block		809	391	743	392
Water Column Depth (ft.)		3600	7157	5470	7210
Anticipated TCW Fluid Category[1]	I	1	1	1	1
	II	0	0	0	0
	III	0	0	1	1
	IV	0	0	1	0
Treatment Type	No Treatment/Tank Storage	1	0	1	0
	Tank Storage	0	1	0	1
	Filtration	0	1	0	0
	Other Treatment	0	0	0	0
Type of Chemical Additives	Corrosion Inhibitor	0	0	1	0
	De-emulsifier	0	1	0	1
	Surfactants	0	1	1	1
	Defoamers	0	1	1	1
	Biocides	0	1	1	1

Notes:
A "0" indicates the variable was absent; a "1" indicates the variable was present.
[1] Category II and IV effluents were not discharged to Gulf of Mexico surface waters during the Year 1 study.

Appendix B.1. Characteristics of TCW Discharges Selected by LHS

Area		Mississippi Canyon	Alaminos Canyon	Green Canyon	Green Canyon
Block		393	857	782	869
Water Column Depth (ft.)		7391	9000	4427	4976
Anticipated TCW Fluid Category[1]	I	1	1	1	1
	II	0	1	0	0
	III	0	1	1	1
	IV	0	0	1	1
Treatment Type	No Treatment/Tank Storage	0	0	1	1
	Tank Storage	1	1	0	0
	Filtration	1	0	0	0
	Other Treatment	0	0	0	0
Type of Chemical Additives	Corrosion Inhibitor	0	1	1	1
	De-emulsifier	1	1	0	0
	Surfactants	1	1	1	1
	Defoamers	1	1	1	1
	Biocides	1	1	1	1

Notes:
A "0" indicates the variable was absent; a "1" indicates the variable was present.
[1] Category II and IV effluents were not discharged to Gulf of Mexico surface waters during the Year 1 study.

Appendix B.1. Characteristics of TCW Discharges Selected by LHS

Area		Mississippi Canyon	Walker Ridge	Mississippi Canyon	Green Canyon
Block		151	678	520	825
Water Column Depth (ft.)		1025	6805	6700	4976
Anticipated TCW Fluid Category[1]	I	1	1	1	1
	II	1	1	0	0
	III	1	1	1	1
	IV	0	1	1	1
Treatment Type	No Treatment/Tank Storage	0	0	1	1
	Tank Storage	1	1	0	0
	Filtration	0	0	0	0
	Other Treatment	0	0	0	0
Type of Chemical Additives	Corrosion Inhibitor	0	0	1	1
	De-emulsifier	0	1	0	0
	Surfactants	0	1	1	1
	Defoamers	0	0	1	1
	Biocides	0	1	1	1

Notes:
A "0" indicates the variable was absent; a "1" indicates the variable was present.
[1] Category II and IV effluents were not discharged to Gulf of Mexico surface waters during the Year 1 study.

Appendix B.1. Characteristics of TCW Discharges Selected by LHS

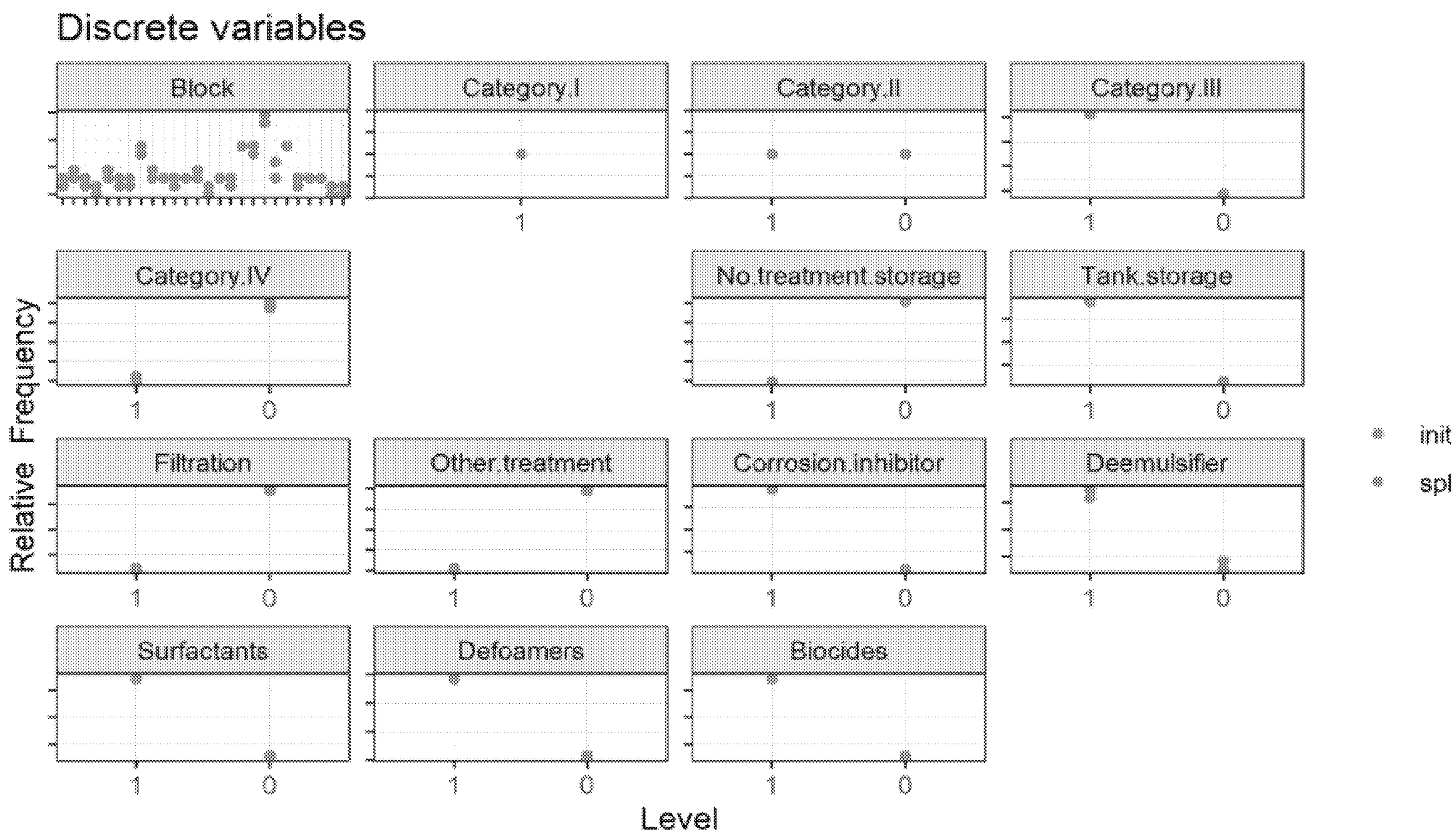
Area		Green Canyon	Alaminos Canyon	Mississippi Canyon	Alaminos Canyon	Green Canyon
Block		826	857	807	857	825
Water Column Depth (ft.)		4976	9000	2940	7815	4976
Anticipated TCW Fluid Category[1]	I	1	1	1	1	1
	II	0	1	1	1	0
	III	1	1	1	1	1
	IV	1	0	0	0	1
Treatment Type	No Treatment/Tank Storage	1	0	0	0	1
	Tank Storage	0	1	1	1	0
	Filtration	0	0	0	0	0
	Other Treatment	0	0	0	0	0
Type of Chemical Additives	Corrosion Inhibitor	1	1	1	1	1
	De-emulsifier	0	1	1	1	0
	Surfactants	1	1	1	1	1
	Defoamers	1	1	1	1	1
	Biocides	1	1	1	1	1

Notes:
A "0" indicates the variable was absent; a "1" indicates the variable was present.
[1] Category II and IV effluents were not discharged to Gulf of Mexico surface waters during the Year 1 study.

Area		Mississippi Canyon
Block		778
Water Column Depth (ft.)		5630
Anticipated TCW Fluid Category[1]	I	1
	II	0
	III	1
	IV	1
Treatment Type	No Treatment/Tank Storage	1
	Tank Storage	0
	Filtration	0
	Other Treatment	0
Type of Chemical Additives	Corrosion Inhibitor	1
	De-emulsifier	0
	Surfactants	1
	Defoamers	1
	Biocides	1

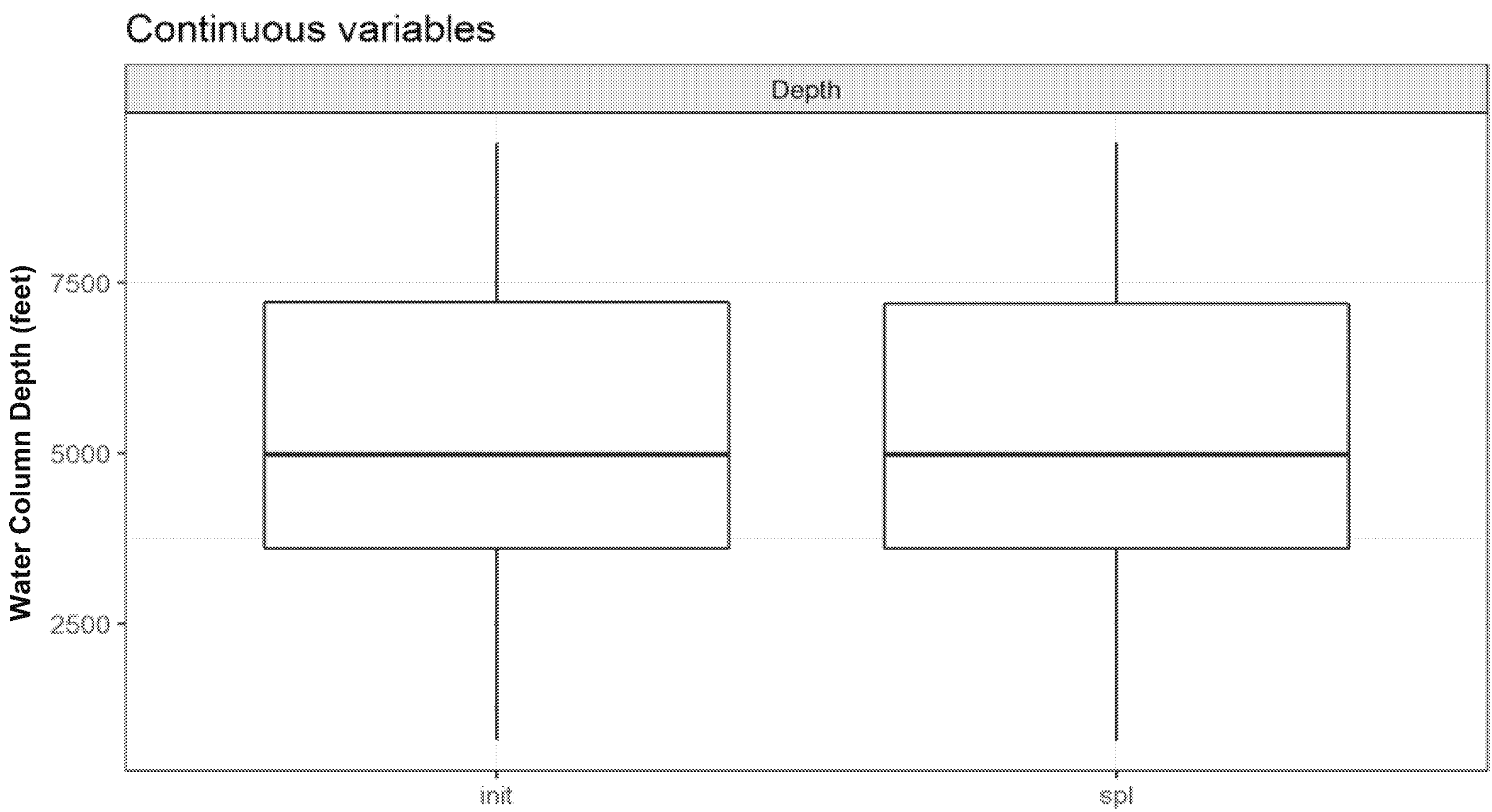
Notes:
A "0" indicates the variable was absent; a "1" indicates the variable was present.
[1] Category II and IV effluents were not discharged to Gulf of Mexico surface waters during the Year 1 study.

Appendix B.2. Comparison of the Sub-sample with the Initial Sample for all LHS Input Variables



Notes:

"init" refers to the initial dataset of 95 discharges; "spl" refers to the selected sub-sample of 34 discharges. The overlap between "init" and "spl" indicates that the 34 discharges are representative of the the larger dataset.



Notes:
"init" refers to the initial dataset of 95 discharges; "spl" refers to the selected sub-sample of 34 discharges.

Appendix C
Supporting Documentation for Statistical Analyses

Appendix C: Supporting Documentation for Statistical Analyses

This appendix presents supporting documentation for statistical analyses. Topics that are discussed are software used, specifics on the box plots, critical values of the Spearman's Ranked Correlation Coefficient (r_s) when n<10, and details of the cluster analysis.

Software Used

Two software programs were used. SYSTAT Ver. 11 (Systat, 2004) was used to prepare boxplots, conduct the Spearman rank-order correlation and Wilcoxon rank-sum analyses, generate the regression plots and fit a quadratic (polynomial) line to the data, and generate the cluster analysis and the resulting dendrogram. ProUCL Ver. 5.1 (USEPA, 2015) was used to calculate the upper confidence limit (UCL) of the mean for the refined Tier 2 acute aquatic toxicity screening. In addition, the Latin hypercube sampling (LHS) evaluation was conducted in "R".

Box Plots

In each box plot, the center vertical line marks the median of the sample. The length of each box shows the range within which the central 50% of the values fall, with the box edges or "hinges" at the first and third quartiles. As defined by SYSTAT, the term "Hspread" is comparable to the interquartile range or midrange and is the difference between the values of the two hinges. The term "fences" is used by SYSTAT to define "outside" and "far outside values". The fences are calculated by SYSTAT as follows:

- Lower inner fence = lower hinge - (1.5 * (Hspread))
- Upper inner fence = upper hinge + (1.5 * (Hspread))
- Lower outer fence = lower hinge - (3 * (Hspread))
- Upper outer fence = upper hinge + (3 * (Hspread))

The whiskers show the range of observed values that fall within the inner fences, i.e., the range of values that fall within 1.5 Hspreads of the hinges. Outside values, i.e., values between the inner and outer fences are plotted with asterisks (*). Values beyond the outer fences, i.e., far outside values, are plotted with empty circles (°).

Critical Values of Spearman Rank-order (r_s)

Statistically significant associations are reported where $p \leq 0.05$. Because *t* is not a good approximation of the sampling distribution of the Spearman r_s when n<10, the following table of non-directional critical values of *r*s was used (Zar, 1984) (**Table C1**).

Table C1. Critical values of the Spearman's Ranked Correlation Coefficient (r_s) when n<10 and non-directional α = 2. Taken from Table B.19 presented in Zar (1984)

α(2):	0.50	0.20	0.10	0.05	0.02	0.01	0.005	0.002	0.001
α(1):	0.25	0.10	0.05	0.025	0.01	0.005	0.0025	0.001	0.0005
n									
4	0.600	1.000	1.000						
5	0.500	0.800	0.900	1.000	1.000				
6	0.371	0.657	0.829	0.886	0.943	1.000	1.000		
7	0.321	0.571	0.714	0.786	0.893	0.929	0.964	1.000	1.000
8	0.310	0.524	0.643	0.738	0.833	0.881	0.905	0.952	0.976
9	0.267	0.483	0.600	0.700	0.783	0.833	0.867	0.917	0.933
10	0.248	0.455	0.564	0.648	0.745	0.794	0.830	0.879	0.903

Cluster Analysis

Cluster analysis is a multivariate procedure that was used to identify natural groupings in the individual WET test endpoint data for Inland silverside minnow and Mysid. Because it is the most sensitive WET test organism, a separate ordination for the Mysid is also presented as a complement to the full ordination presented in the report. The purpose of the Mysid ordination is to illustrate that the ordination with both species is representative of the most sensitive WET test organism.

- **Details of cluster analysis**: Hierarchical and agglomerative cluster analysis was used. Hierarchical clustering produces hierarchical clusters that are displayed in a "tree" or dendrogram. Initially, each TCW effluent sample is considered by SYSTAT as a separate cluster. SYSTAT begins by joining the two "closest" TCW effluent samples as a cluster and continues in a stepwise manner joining a TCW effluent sample with another sample, a sample with a cluster, or a cluster with another cluster until all TCW effluent samples are combined into a single cluster.

  Linkage is used in an ordination to define how distances between clusters are measured. Complete linkage was selected. With the complete linkage option, SYSTAT uses the most distant pair of TCW effluent samples in two clusters to compute between-cluster distances. This method usually yields clusters that are well separated.

  Hierarchical clustering in SYSTAT also allows the user to select the type of distance metric to use between TCW effluent samples when using hierarchical clustering. Euclidean distance was selected. With Euclidean distance, the clustering is computed using normalized Euclidean distance (root mean squared distances). This metric is appropriate for use with quantitative variables. The dendrogram was qualitatively and subjectively "cut" by the user at a Euclidean distance that generated "meaningful" clusters of TCW effluent samples. Several sample-specific factors were considered by the user when identifying clusters: acute aquatic toxicity, well operation, presence and absence of chemical products, and TCW effluent chemistry.

- **Separate ordination of Mysid WET test endpoints**: Similar to the ordination for both species, the dendrogram indicates that TCW Category I and Category III effluents did not ordinate into two separate groups, and that patterns in acute Mysid toxicity are driven by a set of factors more complex than effluent category (**Figure C1**). Nine clusters of effluent samples were identified (Clusters 1-9) that occur along an effluent toxicity gradient. Cluster 1 includes the least toxic sample, which is a Category I effluent with end of pipe treatment. Cluster 9 contains the most toxic TCW effluent samples, which are all Category III effluents, including all the gel samples.

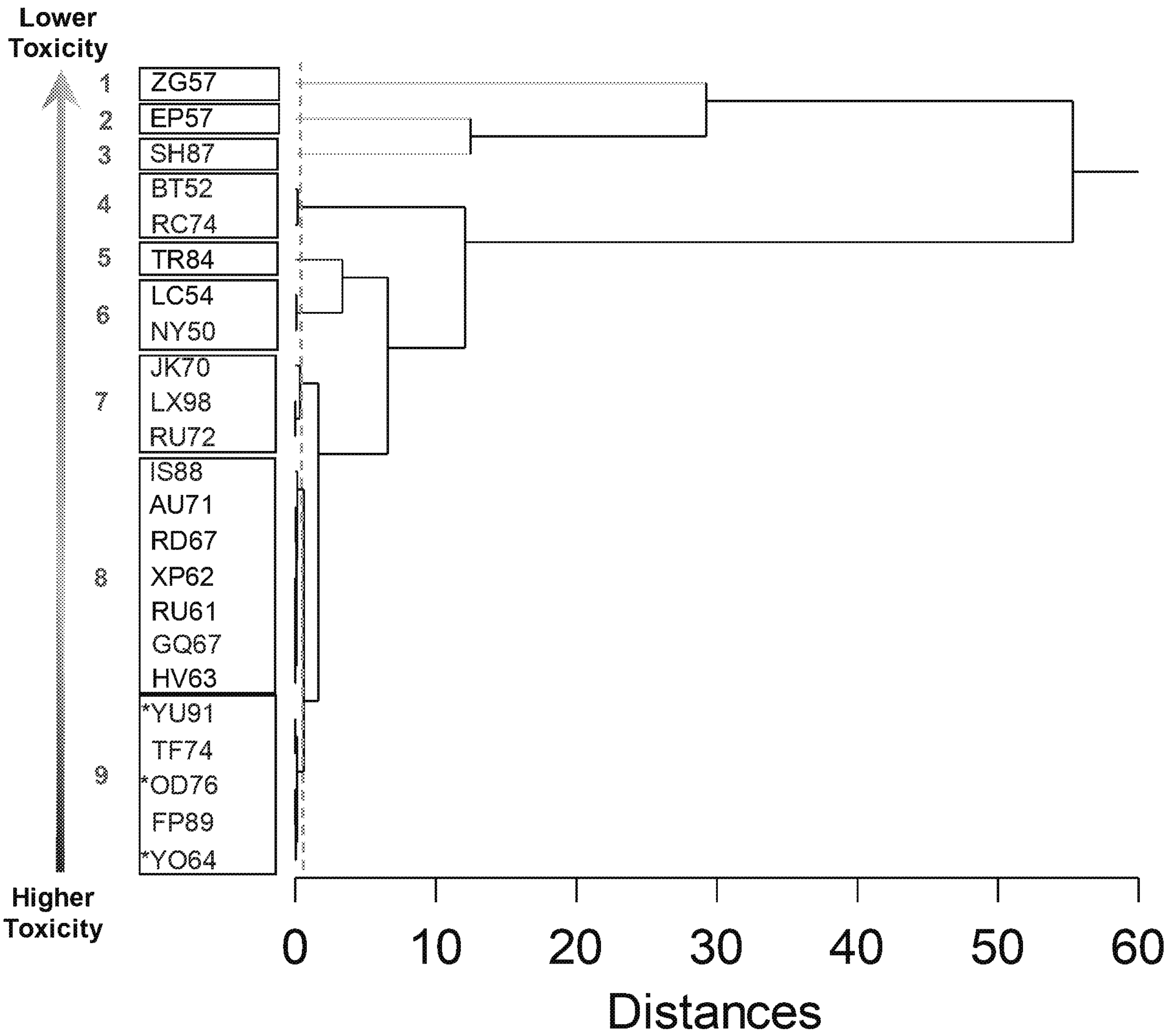


Figure C1. Cluster analysis dendrogram of the Mysid acute WET test endpoints (NOEC, LC25, LOEC, and LC50). TCW Category I effluent samples are presented in black font, TCW Category III samples are in red font, and TCW Category III gel samples are denoted by a (*). The arrow illustrates a whole effluent toxicity gradient; and the vertical dashed blue line indicates where the dendrogram was "cut". The green and red lines are assigned by SYSTAT based on the length of the terminal nodes.

References

U.S. Environmental Protection Agency (Singh, A. and Maichle, R.). 2015. ProUCL Version 5.1 User Guide EPA/600/R-07/041 October 2015 accessed February 2, 2021 at https://www.epa.gov/sites/production/files/2016-05/documents/proucl_5.1_user-guide.pdf

Zar, J.H. 1984. Biostatistical Analysis. 2nd Edition, Prentice-Hall, Inc., Englewood Cliffs, 718 p.

Appendix D
Water Accommodated Fraction (WAF) Aquatic Toxicity Test Procedure and Results

Appendix D: Water Accommodated Fraction (WAF) Aquatic Toxicity Test Procedure and Results

This appendix presents the approach that was used to assess the aquatic toxicity of treatment, completion, and workover (TCW) effluent sample IH80. Category III effluent sample IH80 formed a separate phase when mixed with laboratory control seawater and thus could not be evaluated with standard acute 48-h static renewal WET testing. To characterize the aquatic toxicity of sample IH80, a water accommodated fraction (WAF) test was used. USEPA approved the adoption of the WAF procedure as a departure from the original study plan via email on November 18, 2020.

The term WAF is applied to "*an aqueous test solution containing only the fraction of a substance (or substances) that is dissolved and/or present as a stable dispersion or emulsion*" (Organization for Economic Co-operation and Development [OECD], 2019). The WAF procedure is typically used to address the aquatic toxicity of complex, multi-component substances in crude oil and refined petroleum products. A WAF can contain several dissolved substances, the concentrations of which depend on their water solubility and the mass-to-volume ratio of the preparation (OECD, 2019). Testing was conducted consistent with technical guidance (Ecotoxicology and Toxicology of Chemicals [ECETOC], 1996; OECD, 2019), and the literature (Aurand and Coelho, 2005; Jiang, Huang, Chen, Zeng, and Xu, 2012).

Sample Description

Based on correspondence from the JIP study participant in December 2020, sample IH80 was not discharged to surface water. The sample was inadvertently collected from a holding pit for material that was not intended to be discharged to surface water. As a result, the properties of IH80 are not representative of discharged TCW effluents.

Sample IH80 was collected on April 23, 2020, and the WAF test was conducted from November 11-12, 2020. Hence, the WAF was conducted outside of the WET sample holding time of 36 hours. Based on information provided by the JIP study participant, IH80 consisted of a 12 ppg $CaBr_2$ brine (78%), and two surfactants used as a well cleaner and spacer: "Well Cleaner 1" (17%) and "Well Cleaner 2" (4%). Effluent sample IH80 formed a weakly soluble separate phase when mixed with LCSW and allowed to settle for 24 hours in the laboratory.

Overview of the WAF Procedure

The WAF test procedure involved a preliminary survival range-finding tests, preparation of a stock WAF, sample mixing, settling, WAF recovery, and developing WAF dilutions for use in a definitive aquatic toxicity test. The general WAF experimental design is provided below in **Figure D1**.

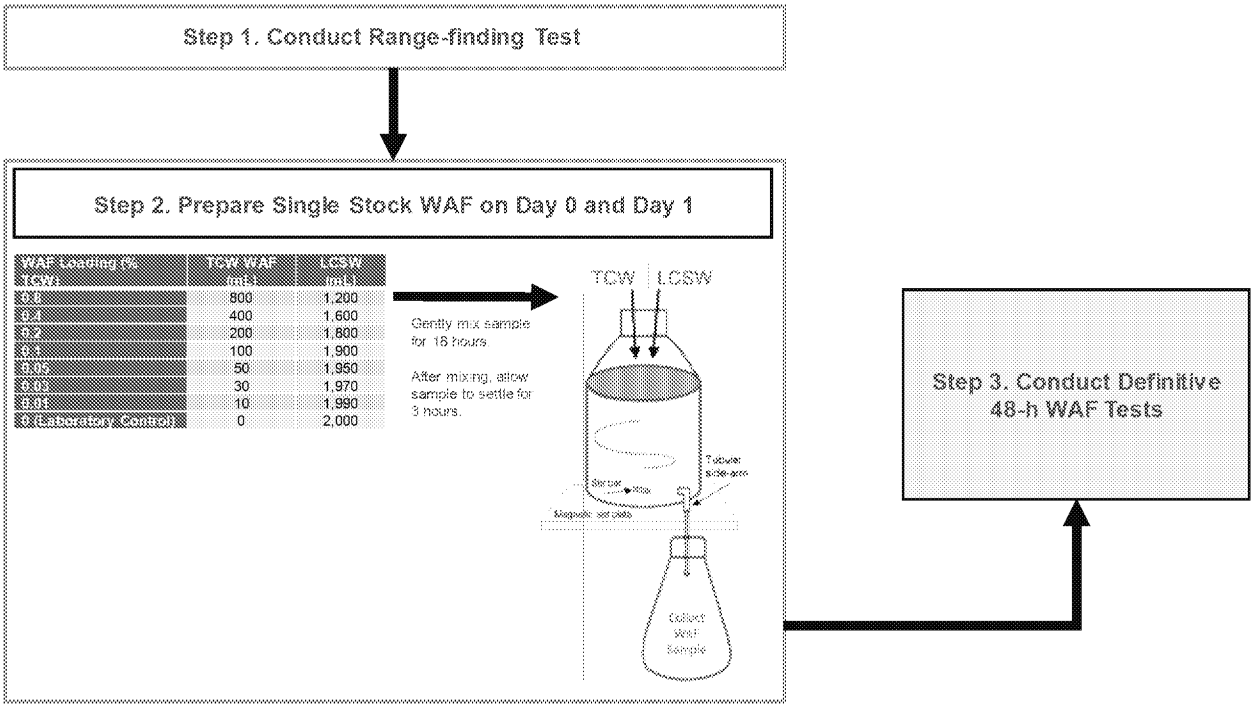


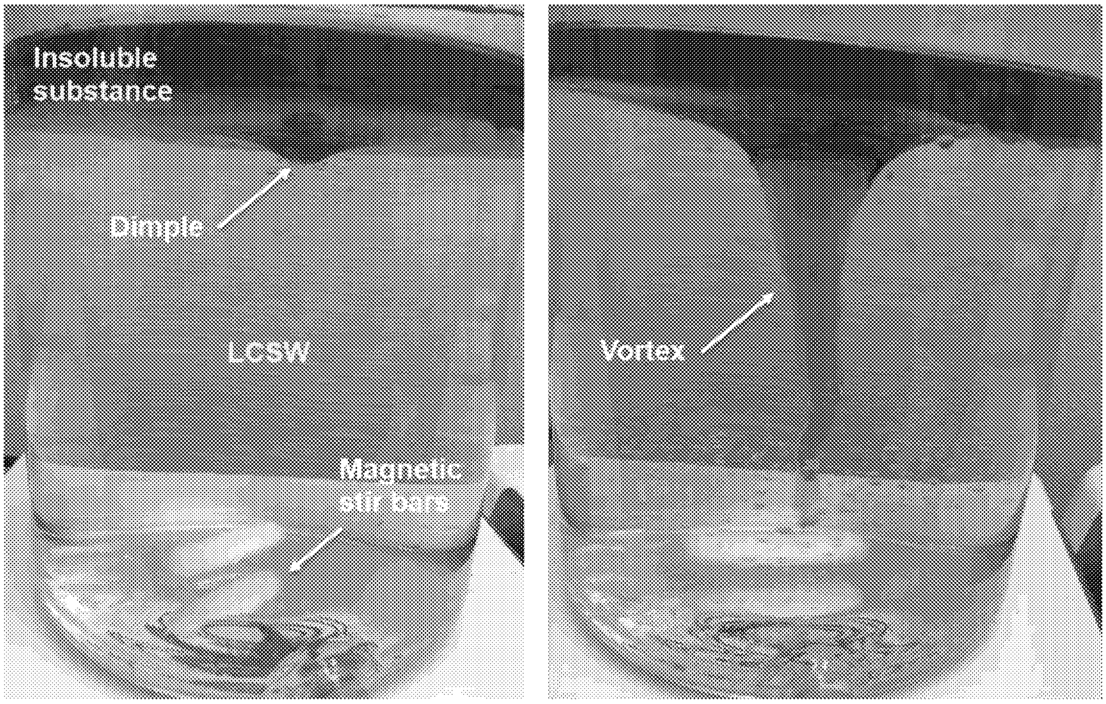
Figure D1. Water accommodated fraction (WAF) experimental design.

Stock WAF Preparation

A stock WAF was prepared with a known mass of TCW sample, mixing the sample, allowing it to settle, recovering the WAF, and developing WAF dilutions for use in a definitive aquatic toxicity test. Sample IH80 was used to prepare a single, 2% TCW by volume stock WAF on Day 0 and Day 1. Each of the 2% TCW WAFs contained 76 milliliters (ml) of IH80 effluent sample and 3,724 mL of LCSW. The preparation of a single stock solution that is diluted for each treatment diverges from technical guidance provided by the OECD (2019) and European Center for Ecotoxicology and Toxicology of Chemicals (ECETOC) (1996). The technical guidance recommends that individual WAFs be prepared. EEUSA deemed the dilution of a single stock WAF sufficient to assess the toxicity of IH80, however, because the product fully dispersed initially. Also, the approach of preparing a single WAF stock solution has been used in other studies (Jiang, Huang, Chen, Zeng, and Xu, 2012).

Sample Mixing

The Day 0 and Day 1 WAFs were prepared in a 4.0-liter (L) glass aspirator bottle, covered, and gently mixed at 340 revolutions per minute (RPM) for 18 hours on a magnetic stir plate (Aurand and Coelho, 2005). When preparing the WAF sample, care was taken to ensure that the mixing rate did not cause the formation of a full "vortex", an emulsion, or suspension of droplets in the aqueous phase. Hence, a slow-stir method such that a small "dimple" formed at the test solution surface was selected consistent with OECD (2019) guidance. An example of a dimple prepared by EEUSA using vegetable oil and red food dye is presented below in **Figure D2**.



Acceptable level of mixing energy - dimple forming at sample surface; no vortex.

Unacceptably high mixing energy - vortex fully formed.

Figure D2. Illustration of acceptable mixing speed for WAF test.

Settling and WAF Sample Recovery

After mixing, the WAF was allowed to settle for three hours (Aurand and Coelho, 2005). At the end of the settling period, 1,800 mL of the 2.0% TCW WAF was recovered from the tubular sidearm outlet of the aspirator bottle. The recovered TCW effluent sample immediately dispersed when mixed with water and remained dispersed.

WAF Loading Rates and Test Dilutions

The WAF loading rate is the quantity of IH80 effluent per unit volume of LCSW used in the preparation of each WAF test medium. A single stock solution of 2.0% TCW WAF was used to prepare the individual WAF dilutions on Day 0 and Day 1. Eight treatments and seven TCW loading rates (0.01%, 0.03%, 0.05%, 0.1%,0.2%, 0.4%, and 0.8% TCW) were prepared daily, in addition to a laboratory control. Individual test chambers were labeled with the test concentration, replicate identification, and an internal laboratory reference number. WAF loadings are provided below in **Table D1.**

Table D1. Water Accommodated Fraction Loadings.		
WAF Loading (% TCW)	**TCW WAF (mL)**	**LCSW (mL)**
0.8	800	1,200
0.4	400	1,600
0.2	200	1,800
0.1	100	1,900
0.05	50	1,950
0.03	30	1,970
0.01	10	1,990
0 (Laboratory Control)	0	2,000

WAF Test Endpoints

Definitive test endpoints are a No Observable Effect Loading (NOEL); a Lowest Observed Effect Loading (LOEL), the 25% Lethal Loading (LL25), and the median Lethal Loading (LL50). The LL25 and LL50 are defined as the lethal WAF loading rate that results in 25% and 50% mortality of exposed organisms, respectively.

WAF Test Results

The 48-h LL50 for Inland silverside minnow exposed to IH80 effluent was 0.03% TCW WAF, and the 48-h LL50 for Mysid was 0.01% TCW WAF. This indicates that the well cleaner products present in IH80 contain substances which are potentially very toxic to aquatic biota. Complete WAF test results are provided below in **Table D2**.

Table D2. 48-h Water Accommodated Fraction (WAF) Aquatic Toxicity Test Results								
Sample	**WAF Test Endpoint (% TCW WAF)**							
	Inland silverside minnow				**Mysid**			
	NOEL	**LL25**	**LOEL**	**LL50**	**NOEL**	**LL25**	**LOEL**	**LL50**
IH80	0.01	0.02	0.03	0.03	<0.01	<0.01	0.01	0.014

References

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Appendix E
ProUCL Documentation

UCL Statistics for Data Sets with Non-Detects

User Selected Options	
Date/Time of Computation	ProUCL 5.111/5/2020 2:05:25 PM
From File	WorkSheet_a.xls
Full Precision	OFF
Confidence Coefficient	95%
Number of Bootstrap Operations	2000

Arsenic

General Statistics

Total Number of Observations	22	Number of Distinct Observations	5
Number of Detects	2	Number of Non-Detects	20
Number of Distinct Detects	2	Number of Distinct Non-Detects	3
Minimum Detect	0.111	Minimum Non-Detect	0.01
Maximum Detect	0.181	Maximum Non-Detect	0.15
Variance Detects	0.00245	Percent Non-Detects	90.91%
Mean Detects	0.146	SD Detects	0.0495
Median Detects	0.146	CV Detects	0.339
Skewness Detects	N/A	Kurtosis Detects	N/A
Mean of Logged Detects	-1.954	SD of Logged Detects	0.346

Warning: Data set has only 2 Detected Values.
This is not enough to compute meaningful or reliable statistics and estimates.

Normal GOF Test on Detects Only
Not Enough Data to Perform GOF Test

Kaplan-Meier (KM) Statistics using Normal Critical Values and other Nonparametric UCLs

KM Mean	0.0226	KM Standard Error of Mean	0.0124
KM SD	0.0407	95% KM (BCA) UCL	N/A
95% KM (t) UCL	0.0439	95% KM (Percentile Bootstrap) UCL	N/A
95% KM (z) UCL	0.0429	95% KM Bootstrap t UCL	N/A
90% KM Chebyshev UCL	0.0597	95% KM Chebyshev UCL	0.0765
97.5% KM Chebyshev UCL	0.0998	99% KM Chebyshev UCL	0.146

Gamma GOF Tests on Detected Observations Only
Not Enough Data to Perform GOF Test

Gamma Statistics on Detected Data Only

k hat (MLE)	17.06	k star (bias corrected MLE)	N/A
Theta hat (MLE)	0.00856	Theta star (bias corrected MLE)	N/A
nu hat (MLE)	68.24	nu star (bias corrected)	N/A
Mean (detects)	0.146		

Estimates of Gamma Parameters using KM Estimates

Mean (KM)	0.0226	SD (KM)	0.0407
Variance (KM)	0.00166	SE of Mean (KM)	0.0124
k hat (KM)	0.308	k star (KM)	0.296
nu hat (KM)	13.55	nu star (KM)	13.04
theta hat (KM)	0.0734	theta star (KM)	0.0763
80% gamma percentile (KM)	0.0345	90% gamma percentile (KM)	0.0667
95% gamma percentile (KM)	0.104	99% gamma percentile (KM)	0.2

Gamma Kaplan-Meier (KM) Statistics

		Adjusted Level of Significance (β)	0.0386
Approximate Chi Square Value (13.04, α)	5.917	Adjusted Chi Square Value (13.04, β)	5.562
95% Gamma Approximate KM-UCL (use when n>=50)	0.0498	95% Gamma Adjusted KM-UCL (use when n<50)	0.053

Lognormal GOF Test on Detected Observations Only
Not Enough Data to Perform GOF Test

Lognormal ROS Statistics Using Imputed Non-Detects

Mean in Original Scale	0.0396	Mean in Log Scale	-3.572
SD in Original Scale	0.0399	SD in Log Scale	0.822
95% t UCL (assumes normality of ROS data)	0.0543	95% Percentile Bootstrap UCL	0.0546
95% BCA Bootstrap UCL	0.0596	95% Bootstrap t UCL	0.067
95% H-UCL (Log ROS)	0.0601		

Arsenic

Statistics using KM estimates on Logged Data and Assuming Lognormal Distribution

KM Mean (logged)	-4.359	KM Geo Mean	0.0128
KM SD (logged)	0.773	95% Critical H Value (KM-Log)	2.288
KM Standard Error of Mean (logged)	0.236	95% H-UCL (KM -Log)	0.0254
KM SD (logged)	0.773	95% Critical H Value (KM-Log)	2.288
KM Standard Error of Mean (logged)	0.236		

DL/2 Statistics

DL/2 Normal		DL/2 Log-Transformed	
Mean in Original Scale	0.0517	Mean in Log Scale	-3.301
SD in Original Scale	0.0375	SD in Log Scale	1.014
95% t UCL (Assumes normality)	0.0654	95% H-Stat UCL	0.11

DL/2 is not a recommended method, provided for comparisons and historical reasons

Nonparametric Distribution Free UCL Statistics

Data do not follow a Discernible Distribution at 5% Significance Level

Suggested UCL to Use

95% KM (t) UCL	0.0439	KM H-UCL	0.0254
95% KM (BCA) UCL	N/A		

Warning: One or more Recommended UCL(s) not available!

Note: Suggestions regarding the selection of a 95% UCL are provided to help the user to select the most appropriate 95% UCL.
Recommendations are based upon data size, data distribution, and skewness.
These recommendations are based upon the results of the simulation studies summarized in Singh, Maichle, and Lee (2006).
However, simulations results will not cover all Real World data sets; for additional insight the user may want to consult a statistician.
However, simulations results will not cover all Real World data sets; for additional insight the user may want to consult a statistician.

UCL Statistics for Uncensored Full Data Sets

User Selected Options	
Date/Time of Computation	ProUCL 5.111/5/2020 2:28:17 PM
From File	WorkSheet.xls
Full Precision	OFF
Confidence Coefficient	95%
Number of Bootstrap Operations	2000

Bromide

General Statistics			
Total Number of Observations	22	Number of Distinct Observations	20
		Number of Missing Observations	0
Minimum	36.7	Mean	598.6
Maximum	8850	Median	46.55
SD	1924	Std. Error of Mean	410.2
Coefficient of Variation	3.214	Skewness	4.173
Normal GOF Test			
Shapiro Wilk Test Statistic	0.329	Shapiro Wilk GOF Test	
5% Shapiro Wilk Critical Value	0.911	Data Not Normal at 5% Significance Level	
Lilliefors Test Statistic	0.433	Lilliefors GOF Test	
5% Lilliefors Critical Value	0.184	Data Not Normal at 5% Significance Level	
Data Not Normal at 5% Significance Level			
Assuming Normal Distribution			
95% Normal UCL		95% UCLs (Adjusted for Skewness)	
95% Student's-t UCL	1304	95% Adjusted-CLT UCL (Chen-1995)	1663
		95% Modified-t UCL (Johnson-1978)	1365
Gamma GOF Test			
A-D Test Statistic	4.853	Anderson-Darling Gamma GOF Test	
5% A-D Critical Value	0.836	Data Not Gamma Distributed at 5% Significance Level	
K-S Test Statistic	0.39	Kolmogorov-Smirnov Gamma GOF Test	
5% K-S Critical Value	0.2	Data Not Gamma Distributed at 5% Significance Level	
Data Not Gamma Distributed at 5% Significance Level			
Gamma Statistics			
k hat (MLE)	0.352	k star (bias corrected MLE)	0.335
Theta hat (MLE)	1699	Theta star (bias corrected MLE)	1789
nu hat (MLE)	15.5	nu star (bias corrected)	14.72
MLE Mean (bias corrected)	598.6	MLE Sd (bias corrected)	1035
		Approximate Chi Square Value (0.05)	7.069
Adjusted Level of Significance	0.0386	Adjusted Chi Square Value	6.675
Assuming Gamma Distribution			
95% Approximate Gamma UCL (use when n>=50))	1247	95% Adjusted Gamma UCL (use when n<50)	1320
Lognormal GOF Test			
Shapiro Wilk Test Statistic	0.644	Shapiro Wilk Lognormal GOF Test	
5% Shapiro Wilk Critical Value	0.911	Data Not Lognormal at 5% Significance Level	
Lilliefors Test Statistic	0.289	Lilliefors Lognormal GOF Test	
5% Lilliefors Critical Value	0.184	Data Not Lognormal at 5% Significance Level	
Data Not Lognormal at 5% Significance Level			
Lognormal Statistics			
Minimum of Logged Data	3.603	Mean of logged Data	4.488
Maximum of Logged Data	9.088	SD of logged Data	1.466
Assuming Lognormal Distribution			
95% H-UCL	744.8	90% Chebyshev (MVUE) UCL	507.2
95% Chebyshev (MVUE) UCL	629.2	97.5% Chebyshev (MVUE) UCL	798.5
99% Chebyshev (MVUE) UCL	1131		
Nonparametric Distribution Free UCL Statistics			
Data do not follow a Discernible Distribution (0.05)			
Nonparametric Distribution Free UCLs			
95% CLT UCL	1273	95% Jackknife UCL	1304

Bromide

95% Standard Bootstrap UCL	1240	95% Bootstrap-t UCL	11207
95% Hall's Bootstrap UCL	11179	95% Percentile Bootstrap UCL	1386
95% BCA Bootstrap UCL	2090		
90% Chebyshev(Mean, Sd) UCL	1829	95% Chebyshev(Mean, Sd) UCL	2386
97.5% Chebyshev(Mean, Sd) UCL	3160	99% Chebyshev(Mean, Sd) UCL	4680

Suggested UCL to Use

95% Chebyshev (Mean, Sd) UCL	2386

Note: Suggestions regarding the selection of a 95% UCL are provided to help the user to select the most appropriate 95% UCL.
Recommendations are based upon data size, data distribution, and skewness.
These recommendations are based upon the results of the simulation studies summarized in Singh, Maichle, and Lee (2006).
However, simulations results will not cover all Real World data sets; for additional insight the user may want to consult a statistician.

Calcium

General Statistics			
Total Number of Observations	22	Number of Distinct Observations	22
		Number of Missing Observations	0
Minimum	220	Mean	461.4
Maximum	2370	Median	281
SD	465.4	Std. Error of Mean	99.23
Coefficient of Variation	1.009	Skewness	3.612
Normal GOF Test			
Shapiro Wilk Test Statistic	0.511	Shapiro Wilk GOF Test	
5% Shapiro Wilk Critical Value	0.911	Data Not Normal at 5% Significance Level	
Lilliefors Test Statistic	0.302	Lilliefors GOF Test	
5% Lilliefors Critical Value	0.184	Data Not Normal at 5% Significance Level	
Data Not Normal at 5% Significance Level			
Assuming Normal Distribution			
95% Normal UCL		95% UCLs (Adjusted for Skewness)	
95% Student's-t UCL	632.2	95% Adjusted-CLT UCL (Chen-1995)	706.3
		95% Modified-t UCL (Johnson-1978)	644.9
Gamma GOF Test			
A-D Test Statistic	2.518	Anderson-Darling Gamma GOF Test	
5% A-D Critical Value	0.754	Data Not Gamma Distributed at 5% Significance Level	
K-S Test Statistic	0.296	Kolmogorov-Smirnov Gamma GOF Test	
5% K-S Critical Value	0.187	Data Not Gamma Distributed at 5% Significance Level	
Data Not Gamma Distributed at 5% Significance Level			
Gamma Statistics			
k hat (MLE)	2.365	k star (bias corrected MLE)	2.073
Theta hat (MLE)	195.1	Theta star (bias corrected MLE)	222.6
nu hat (MLE)	104.1	nu star (bias corrected)	91.22
MLE Mean (bias corrected)	461.4	MLE Sd (bias corrected)	320.5
		Approximate Chi Square Value (0.05)	70.19
Adjusted Level of Significance	0.0386	Adjusted Chi Square Value	68.82
Assuming Gamma Distribution			
95% Approximate Gamma UCL (use when n>=50))	599.6	95% Adjusted Gamma UCL (use when n<50)	611.6
Lognormal GOF Test			
Shapiro Wilk Test Statistic	0.76	Shapiro Wilk Lognormal GOF Test	
5% Shapiro Wilk Critical Value	0.911	Data Not Lognormal at 5% Significance Level	
Lilliefors Test Statistic	0.282	Lilliefors Lognormal GOF Test	
5% Lilliefors Critical Value	0.184	Data Not Lognormal at 5% Significance Level	
Data Not Lognormal at 5% Significance Level			
Lognormal Statistics			
Minimum of Logged Data	5.394	Mean of logged Data	5.908
Maximum of Logged Data	7.771	SD of logged Data	0.582
Assuming Lognormal Distribution			
95% H-UCL	567.2	90% Chebyshev (MVUE) UCL	600.9
95% Chebyshev (MVUE) UCL	677.4	97.5% Chebyshev (MVUE) UCL	783.6
99% Chebyshev (MVUE) UCL	992.2		
Nonparametric Distribution Free UCL Statistics			
Data do not follow a Discernible Distribution (0.05)			
Nonparametric Distribution Free UCLs			
95% CLT UCL	624.6	95% Jackknife UCL	632.2
95% Standard Bootstrap UCL	619.4	95% Bootstrap-t UCL	896.8
95% Hall's Bootstrap UCL	1169	95% Percentile Bootstrap UCL	654.4
95% BCA Bootstrap UCL	745.7		
90% Chebyshev(Mean, Sd) UCL	759.1	95% Chebyshev(Mean, Sd) UCL	894
97.5% Chebyshev(Mean, Sd) UCL	1081	99% Chebyshev(Mean, Sd) UCL	1449
Suggested UCL to Use			
95% Chebyshev (Mean, Sd) UCL	894		

Note: Suggestions regarding the selection of a 95% UCL are provided to help the user to select the most appropriate 95% UCL.
Recommendations are based upon data size, data distribution, and skewness.

Calcium

These recommendations are based upon the results of the simulation studies summarized in Singh, Maichle, and Lee (2006).
However, simulations results will not cover all Real World data sets; for additional insight the user may want to consult a statistician.

Copper

General Statistics			
Total Number of Observations	22	Number of Distinct Observations	13
Number of Detects	13	Number of Non-Detects	9
Number of Distinct Detects	11	Number of Distinct Non-Detects	2
Minimum Detect	0.006	Minimum Non-Detect	0.03
Maximum Detect	0.055	Maximum Non-Detect	0.05
Variance Detects	2.6210E-4	Percent Non-Detects	40.91%
Mean Detects	0.0315	SD Detects	0.0162
Median Detects	0.035	CV Detects	0.515
Skewness Detects	-0.513	Kurtosis Detects	-1.026
Mean of Logged Detects	-3.661	SD of Logged Detects	0.755
Normal GOF Test on Detects Only			
Shapiro Wilk Test Statistic	0.894	Shapiro Wilk GOF Test	
5% Shapiro Wilk Critical Value	0.866	Detected Data appear Normal at 5% Significance Level	
Lilliefors Test Statistic	0.255	Lilliefors GOF Test	
5% Lilliefors Critical Value	0.234	Detected Data Not Normal at 5% Significance Level	
Detected Data appear Approximate Normal at 5% Significance Level			
Kaplan-Meier (KM) Statistics using Normal Critical Values and other Nonparametric UCLs			
KM Mean	0.0232	KM Standard Error of Mean	0.00381
KM SD	0.0162	95% KM (BCA) UCL	0.0297
95% KM (t) UCL	0.0298	95% KM (Percentile Bootstrap) UCL	0.0296
95% KM (z) UCL	0.0295	95% KM Bootstrap t UCL	0.0302
90% KM Chebyshev UCL	0.0347	95% KM Chebyshev UCL	0.0398
97.5% KM Chebyshev UCL	0.047	99% KM Chebyshev UCL	0.0611
Gamma GOF Tests on Detected Observations Only			
A-D Test Statistic	1.068	Anderson-Darling GOF Test	
5% A-D Critical Value	0.741	Detected Data Not Gamma Distributed at 5% Significance Level	
K-S Test Statistic	0.323	Kolmogorov-Smirnov GOF	
5% K-S Critical Value	0.239	Detected Data Not Gamma Distributed at 5% Significance Level	
Detected Data Not Gamma Distributed at 5% Significance Level			
Gamma Statistics on Detected Data Only			
k hat (MLE)	2.629	k star (bias corrected MLE)	2.074
Theta hat (MLE)	0.012	Theta star (bias corrected MLE)	0.0152
nu hat (MLE)	68.36	nu star (bias corrected)	53.92
Mean (detects)	0.0315		
Gamma ROS Statistics using Imputed Non-Detects			
GROS may not be used when data set has > 50% NDs with many tied observations at multiple DLs			
GROS may not be used when kstar of detects is small such as <1.0, especially when the sample size is small (e.g., <15-20)			
For such situations, GROS method may yield incorrect values of UCLs and BTVs			
This is especially true when the sample size is small.			
For gamma distributed detected data, BTVs and UCLs may be computed using gamma distribution on KM estimates			
Minimum	0.006	Mean	0.0252
Maximum	0.055	Median	0.0209
SD	0.0148	CV	0.587
k hat (MLE)	2.807	k star (bias corrected MLE)	2.455
Theta hat (MLE)	0.00897	Theta star (bias corrected MLE)	0.0103
nu hat (MLE)	123.5	nu star (bias corrected)	108
Adjusted Level of Significance (β)	0.0386		
Approximate Chi Square Value (108.01, α)	85.03	Adjusted Chi Square Value (108.01, β)	83.51
95% Gamma Approximate UCL (use when n>=50)	0.032	95% Gamma Adjusted UCL (use when n<50)	0.0326
Estimates of Gamma Parameters using KM Estimates			
Mean (KM)	0.0232	SD (KM)	0.0162
Variance (KM)	2.6337E-4	SE of Mean (KM)	0.00381
k hat (KM)	2.046	k star (KM)	1.797
nu hat (KM)	90.03	nu star (KM)	79.08
theta hat (KM)	0.0113	theta star (KM)	0.0129
80% gamma percentile (KM)	0.0352	90% gamma percentile (KM)	0.0463
95% gamma percentile (KM)	0.057	99% gamma percentile (KM)	0.0808
Gamma Kaplan-Meier (KM) Statistics			
Approximate Chi Square Value (79.08, α)	59.59	Adjusted Chi Square Value (79.08, β)	58.33
95% Gamma Approximate KM-UCL (use when n>=50)	0.0308	95% Gamma Adjusted KM-UCL (use when n<50)	0.0315
Lognormal GOF Test on Detected Observations Only			

Copper

Shapiro Wilk Test Statistic	0.802	Shapiro Wilk GOF Test	
5% Shapiro Wilk Critical Value	0.866	Detected Data Not Lognormal at 5% Significance Level	
Lilliefors Test Statistic	0.337	Lilliefors GOF Test	
5% Lilliefors Critical Value	0.234	Detected Data Not Lognormal at 5% Significance Level	
Detected Data Not Lognormal at 5% Significance Level			
Lognormal ROS Statistics Using Imputed Non-Detects			
Mean in Original Scale	0.0236	Mean in Log Scale	-3.991
SD in Original Scale	0.0158	SD in Log Scale	0.747
95% t UCL (assumes normality of ROS data)	0.0294	95% Percentile Bootstrap UCL	0.029
95% BCA Bootstrap UCL	0.0295	95% Bootstrap t UCL	0.0305
95% H-UCL (Log ROS)	0.0353		
Statistics using KM estimates on Logged Data and Assuming Lognormal Distribution			
KM Mean (logged)	-4.053	KM Geo Mean	0.0174
KM SD (logged)	0.791	95% Critical H Value (KM-Log)	2.309
KM Standard Error of Mean (logged)	0.202	95% H-UCL (KM -Log)	0.0354
KM SD (logged)	0.791	95% Critical H Value (KM-Log)	2.309
KM Standard Error of Mean (logged)	0.202		
DL/2 Statistics			
DL/2 Normal		DL/2 Log-Transformed	
Mean in Original Scale	0.0252	Mean in Log Scale	-3.858
SD in Original Scale	0.0146	SD in Log Scale	0.629
95% t UCL (Assumes normality)	0.0305	95% H-Stat UCL	0.0344
DL/2 is not a recommended method, provided for comparisons and historical reasons			
Nonparametric Distribution Free UCL Statistics			
Detected Data appear Approximate Normal Distributed at 5% Significance Level			
Suggested UCL to Use			
95% KM (t) UCL	0.0298		

When a data set follows an approximate (e.g., normal) distribution passing one of the GOF test
When applicable, it is suggested to use a UCL based upon a distribution (e.g., gamma) passing both GOF tests in ProUCL

Note: Suggestions regarding the selection of a 95% UCL are provided to help the user to select the most appropriate 95% UCL.
Recommendations are based upon data size, data distribution, and skewness.
These recommendations are based upon the results of the simulation studies summarized in Singh, Maichle, and Lee (2006).
However, simulations results will not cover all Real World data sets; for additional insight the user may want to consult a statistician.

Zinc

General Statistics

Total Number of Observations	22	Number of Distinct Observations	8
Number of Detects	7	Number of Non-Detects	15
Number of Distinct Detects	7	Number of Distinct Non-Detects	1
Minimum Detect	0.014	Minimum Non-Detect	0.1
Maximum Detect	0.226	Maximum Non-Detect	0.1
Variance Detects	0.00565	Percent Non-Detects	68.18%
Mean Detects	0.107	SD Detects	0.0752
Median Detects	0.105	CV Detects	0.7
Skewness Detects	0.172	Kurtosis Detects	-0.469
Mean of Logged Detects	-2.591	SD of Logged Detects	1.069

Normal GOF Test on Detects Only

Shapiro Wilk Test Statistic	0.947	Shapiro Wilk GOF Test	
5% Shapiro Wilk Critical Value	0.803	Detected Data appear Normal at 5% Significance Level	
Lilliefors Test Statistic	0.163	Lilliefors GOF Test	
5% Lilliefors Critical Value	0.304	Detected Data appear Normal at 5% Significance Level	

Detected Data appear Normal at 5% Significance Level

Kaplan-Meier (KM) Statistics using Normal Critical Values and other Nonparametric UCLs

KM Mean	0.0628	KM Standard Error of Mean	0.0212
KM SD	0.0577	95% KM (BCA) UCL	0.109
95% KM (t) UCL	0.0993	95% KM (Percentile Bootstrap) UCL	0.108
95% KM (z) UCL	0.0977	95% KM Bootstrap t UCL	0.125
90% KM Chebyshev UCL	0.126	95% KM Chebyshev UCL	0.155
97.5% KM Chebyshev UCL	0.195	99% KM Chebyshev UCL	0.274

Gamma GOF Tests on Detected Observations Only

A-D Test Statistic	0.465	Anderson-Darling GOF Test	
5% A-D Critical Value	0.721	Detected data appear Gamma Distributed at 5% Significance Level	
K-S Test Statistic	0.249	Kolmogorov-Smirnov GOF	
5% K-S Critical Value	0.317	Detected data appear Gamma Distributed at 5% Significance Level	

Detected data appear Gamma Distributed at 5% Significance Level

Gamma Statistics on Detected Data Only

k hat (MLE)	1.534	k star (bias corrected MLE)	0.972
Theta hat (MLE)	0.07	Theta star (bias corrected MLE)	0.111
nu hat (MLE)	21.48	nu star (bias corrected)	13.61
Mean (detects)	0.107		

Gamma ROS Statistics using Imputed Non-Detects

GROS may not be used when data set has > 50% NDs with many tied observations at multiple DLs

GROS may not be used when kstar of detects is small such as <1.0, especially when the sample size is small (e.g., <15-20)

For such situations, GROS method may yield incorrect values of UCLs and BTVs

This is especially true when the sample size is small.

For gamma distributed detected data, BTVs and UCLs may be computed using gamma distribution on KM estimates

Minimum	0.01	Mean	0.0615
Maximum	0.226	Median	0.0442
SD	0.0566	CV	0.92
k hat (MLE)	1.313	k star (bias corrected MLE)	1.164
Theta hat (MLE)	0.0469	Theta star (bias corrected MLE)	0.0529
nu hat (MLE)	57.76	nu star (bias corrected)	51.22
Adjusted Level of Significance (β)	0.0386		
Approximate Chi Square Value (51.22, α)	35.78	Adjusted Chi Square Value (51.22, β)	34.82
95% Gamma Approximate UCL (use when n>=50)	0.0881	95% Gamma Adjusted UCL (use when n<50)	0.0905

Estimates of Gamma Parameters using KM Estimates

Mean (KM)	0.0628	SD (KM)	0.0577
Variance (KM)	0.00333	SE of Mean (KM)	0.0212
k hat (KM)	1.186	k star (KM)	1.055
nu hat (KM)	52.18	nu star (KM)	46.4
theta hat (KM)	0.053	theta star (KM)	0.0596
80% gamma percentile (KM)	0.101	90% gamma percentile (KM)	0.143
95% gamma percentile (KM)	0.185	99% gamma percentile (KM)	0.282

Gamma Kaplan-Meier (KM) Statistics

Approximate Chi Square Value (46.40, α)	31.77	Adjusted Chi Square Value (46.40, β)	30.87
95% Gamma Approximate KM-UCL (use when n>=50)	0.0917	95% Gamma Adjusted KM-UCL (use when n<50)	0.0944

Lognormal GOF Test on Detected Observations Only

Zinc

Shapiro Wilk Test Statistic	0.848	Shapiro Wilk GOF Test	
5% Shapiro Wilk Critical Value	0.803	Detected Data appear Lognormal at 5% Significance Level	
Lilliefors Test Statistic	0.29	Lilliefors GOF Test	
5% Lilliefors Critical Value	0.304	Detected Data appear Lognormal at 5% Significance Level	
Detected Data appear Lognormal at 5% Significance Level			
Lognormal ROS Statistics Using Imputed Non-Detects			
Mean in Original Scale	0.0569	Mean in Log Scale	-3.301
SD in Original Scale	0.0564	SD in Log Scale	0.969
95% t UCL (assumes normality of ROS data)	0.0776	95% Percentile Bootstrap UCL	0.0775
95% BCA Bootstrap UCL	0.0809	95% Bootstrap t UCL	0.0854
95% H-UCL (Log ROS)	0.101		
Statistics using KM estimates on Logged Data and Assuming Lognormal Distribution			
KM Mean (logged)	-3.226	KM Geo Mean	0.0397
KM SD (logged)	0.977	95% Critical H Value (KM-Log)	2.551
KM Standard Error of Mean (logged)	0.442	95% H-UCL (KM -Log)	0.11
KM SD (logged)	0.977	95% Critical H Value (KM-Log)	2.551
KM Standard Error of Mean (logged)	0.442		
DL/2 Statistics			
DL/2 Normal		DL/2 Log-Transformed	
Mean in Original Scale	0.0683	Mean in Log Scale	-2.867
SD in Original Scale	0.0486	SD in Log Scale	0.603
95% t UCL (Assumes normality)	0.0861	95% H-Stat UCL	0.0899
DL/2 is not a recommended method, provided for comparisons and historical reasons			
Nonparametric Distribution Free UCL Statistics			
Detected Data appear Normal Distributed at 5% Significance Level			
Suggested UCL to Use			
95% KM (t) UCL	0.0993		

Note: Suggestions regarding the selection of a 95% UCL are provided to help the user to select the most appropriate 95% UCL.
Recommendations are based upon data size, data distribution, and skewness.
These recommendations are based upon the results of the simulation studies summarized in Singh, Maichle, and Lee (2006).
However, simulations results will not cover all Real World data sets; for additional insight the user may want to consult a statistician.

UCL Statistics for Data Sets with Non-Detects

User Selected Options	
Date/Time of Computation	ProUCL 5.111/5/2020 2:19:18 PM
From File	WorkSheet_a.xls
Full Precision	OFF
Confidence Coefficient	95%
Number of Bootstrap Operations	2000

Arsenic

General Statistics

Total Number of Observations	22	Number of Distinct Observations	5
Number of Detects	2	Number of Non-Detects	20
Number of Distinct Detects	2	Number of Distinct Non-Detects	3
Minimum Detect	0.0139	Minimum Non-Detect	0.01
Maximum Detect	0.288	Maximum Non-Detect	0.15
Variance Detects	0.0376	Percent Non-Detects	90.91%
Mean Detects	0.151	SD Detects	0.194
Median Detects	0.151	CV Detects	1.284
Skewness Detects	N/A	Kurtosis Detects	N/A
Mean of Logged Detects	-2.76	SD of Logged Detects	2.143

Warning: Data set has only 2 Detected Values.
This is not enough to compute meaningful or reliable statistics and estimates.

Normal GOF Test on Detects Only
Not Enough Data to Perform GOF Test

Kaplan-Meier (KM) Statistics using Normal Critical Values and other Nonparametric UCLs

KM Mean	0.0236	KM Standard Error of Mean	0.0174
KM SD	0.0577	95% KM (BCA) UCL	N/A
95% KM (t) UCL	0.0536	95% KM (Percentile Bootstrap) UCL	N/A
95% KM (z) UCL	0.0522	95% KM Bootstrap t UCL	N/A
90% KM Chebyshev UCL	0.0759	95% KM Chebyshev UCL	0.0996
97.5% KM Chebyshev UCL	0.132	99% KM Chebyshev UCL	0.197

Gamma GOF Tests on Detected Observations Only
Not Enough Data to Perform GOF Test

Gamma Statistics on Detected Data Only

k hat (MLE)	0.696	k star (bias corrected MLE)	N/A
Theta hat (MLE)	0.217	Theta star (bias corrected MLE)	N/A
nu hat (MLE)	2.783	nu star (bias corrected)	N/A
Mean (detects)	0.151		

Estimates of Gamma Parameters using KM Estimates

Mean (KM)	0.0236	SD (KM)	0.0577
Variance (KM)	0.00333	footer	0.0174
k hat (KM)	0.167	k star (KM)	0.174
nu hat (KM)	7.333	nu star (KM)	7.667
Arsenic			
theta hat (KM)	0.141	theta star (KM)	0.135
80% gamma percentile (KM)	0.0286	90% gamma percentile (KM)	0.0709
95% gamma percentile (KM)	0.126	99% gamma percentile (KM)	0.28

Gamma Kaplan-Meier (KM) Statistics

		Adjusted Level of Significance (β)	0.0386
Approximate Chi Square Value (7.67, α)	2.543	Adjusted Chi Square Value (7.67, β)	2.328
95% Gamma Approximate KM-UCL (use when n>=50)	0.071	95% Gamma Adjusted KM-UCL (use when n<50)	0.0776

Lognormal GOF Test on Detected Observations Only
Not Enough Data to Perform GOF Test

Lognormal ROS Statistics Using Imputed Non-Detects

Mean in Original Scale	0.017	Mean in Log Scale	-7.103
SD in Original Scale	0.061	SD in Log Scale	2.578
95% t UCL (assumes normality of ROS data)	0.0394	95% Percentile Bootstrap UCL	0.0428
95% BCA Bootstrap UCL	0.0572	95% Bootstrap t UCL	0.235
95% H-UCL (Log ROS)	0.429		

Statistics using KM estimates on Logged Data and Assuming Lognormal Distribution

Arsenic

KM Mean (logged)	-4.374	KM Geo Mean	0.0126
KM SD (logged)	0.697	95% Critical H Value (KM-Log)	2.199
KM Standard Error of Mean (logged)	0.227	95% H-UCL (KM -Log)	0.0224
KM SD (logged)	0.697	95% Critical H Value (KM-Log)	2.199
KM Standard Error of Mean (logged)	0.227		

DL/2 Statistics

DL/2 Normal		DL/2 Log-Transformed	
Mean in Original Scale	0.0542	Mean in Log Scale	-3.27
SD in Original Scale	0.0553	SD in Log Scale	0.954
95% t UCL (Assumes normality)	0.0745	95% H-Stat UCL	0.101

DL/2 is not a recommended method, provided for comparisons and historical reasons

Nonparametric Distribution Free UCL Statistics
Data do not follow a Discernible Distribution at 5% Significance Level

Suggested UCL to Use

97.5% KM (Chebyshev) UCL	0.132

Note: Suggestions regarding the selection of a 95% UCL are provided to help the user to select the most appropriate 95% UCL.
Recommendations are based upon data size, data distribution, and skewness.
These recommendations are based upon the results of the simulation studies summarized in Singh, Maichle, and Lee (2006).
However, simulations results will not cover all Real World data sets; for additional insight the user may want to consult a statistician.

Calcium

General Statistics

Total Number of Observations	22	Number of Distinct Observations	20
		Number of Missing Observations	0
Minimum	216	Mean	448
Maximum	2140	Median	284
SD	415.9	Std. Error of Mean	88.66
Coefficient of Variation	0.928	Skewness	3.536

Normal GOF Test

Shapiro Wilk Test Statistic	0.526	Shapiro Wilk GOF Test	
5% Shapiro Wilk Critical Value	0.911	Data Not Normal at 5% Significance Level	
Lilliefors Test Statistic	0.319	Lilliefors GOF Test	
5% Lilliefors Critical Value	0.184	Data Not Normal at 5% Significance Level	

Data Not Normal at 5% Significance Level

Assuming Normal Distribution

95% Normal UCL		95% UCLs (Adjusted for Skewness)	
95% Student's-t UCL	600.6	95% Adjusted-CLT UCL (Chen-1995)	665.3
		95% Modified-t UCL (Johnson-1978)	611.7

Gamma GOF Test

A-D Test Statistic	2.442	Anderson-Darling Gamma GOF Test	
5% A-D Critical Value	0.752	Data Not Gamma Distributed at 5% Significance Level	
K-S Test Statistic	0.27	Kolmogorov-Smirnov Gamma GOF Test	
5% K-S Critical Value	0.187	Data Not Gamma Distributed at 5% Significance Level	

Data Not Gamma Distributed at 5% Significance Level

Gamma Statistics

k hat (MLE)	2.673	k star (bias corrected MLE)	2.339
Theta hat (MLE)	167.6	Theta star (bias corrected MLE)	191.6
nu hat (MLE)	117.6	nu star (bias corrected)	102.9
MLE Mean (bias corrected)	448	MLE Sd (bias corrected)	293
		Approximate Chi Square Value (0.05)	80.49
Adjusted Level of Significance	0.0386	Adjusted Chi Square Value	79.01

Assuming Gamma Distribution

95% Approximate Gamma UCL (use when n>=50))	572.8	95% Adjusted Gamma UCL (use when n<50)	583.5

Lognormal GOF Test

Shapiro Wilk Test Statistic	0.771	Shapiro Wilk Lognormal GOF Test	
5% Shapiro Wilk Critical Value	0.911	Data Not Lognormal at 5% Significance Level	
Lilliefors Test Statistic	0.251	Lilliefors Lognormal GOF Test	
5% Lilliefors Critical Value	0.184	Data Not Lognormal at 5% Significance Level	

Data Not Lognormal at 5% Significance Level

Lognormal Statistics

Minimum of Logged Data	5.375	Mean of logged Data	5.906
Maximum of Logged Data	7.669	SD of logged Data	0.549

Assuming Lognormal Distribution

Calcium

95% H-UCL	545.7	90% Chebyshev (MVUE) UCL	579.6
95% Chebyshev (MVUE) UCL	650.1	97.5% Chebyshev (MVUE) UCL	748.1
99% Chebyshev (MVUE) UCL	940.4		

Nonparametric Distribution Free UCL Statistics

Data do not follow a Discernible Distribution (0.05)

Nonparametric Distribution Free UCLs

95% CLT UCL	593.9	95% Jackknife UCL	600.6
95% Standard Bootstrap UCL	593.6	95% Bootstrap-t UCL	805.2
95% Hall's Bootstrap UCL	1078	95% Percentile Bootstrap UCL	599.2
95% BCA Bootstrap UCL	693.7		
90% Chebyshev(Mean, Sd) UCL	714	95% Chebyshev(Mean, Sd) UCL	834.5
97.5% Chebyshev(Mean, Sd) UCL	1002	99% Chebyshev(Mean, Sd) UCL	1330

Suggested UCL to Use

95% Chebyshev (Mean, Sd) UCL	834.5

Note: Suggestions regarding the selection of a 95% UCL are provided to help the user to select the most appropriate 95% UCL.
Recommendations are based upon data size, data distribution, and skewness.

Calcium
These recommendations are based upon the results of the simulation studies summarized in Singh, Maichle, and Lee (2006).
However, simulations results will not cover all Real World data sets; for additional insight the user may want to consult a statistician.

Copper

General Statistics

Total Number of Observations	22	Number of Distinct Observations	7
Number of Detects	5	Number of Non-Detects	17
Number of Distinct Detects	5	Number of Distinct Non-Detects	2
Minimum Detect	0.0058	Minimum Non-Detect	0.03
Maximum Detect	0.046	Maximum Non-Detect	0.05
Variance Detects	2.7385E-4	Percent Non-Detects	77.27%
Mean Detects	0.0169	SD Detects	0.0165
Median Detects	0.0117	CV Detects	0.98
Skewness Detects	2.057	Kurtosis Detects	4.37
Mean of Logged Detects	-4.374	SD of Logged Detects	0.795

Normal GOF Test on Detects Only

Shapiro Wilk Test Statistic	0.718	Shapiro Wilk GOF Test	
5% Shapiro Wilk Critical Value	0.762	Detected Data Not Normal at 5% Significance Level	
Lilliefors Test Statistic	0.388	Lilliefors GOF Test	
5% Lilliefors Critical Value	0.343	Detected Data Not Normal at 5% Significance Level	

Detected Data Not Normal at 5% Significance Level

Kaplan-Meier (KM) Statistics using Normal Critical Values and other Nonparametric UCLs

KM Mean	0.0157	KM Standard Error of Mean	0.00635
KM SD	0.0138	95% KM (BCA) UCL	0.0293
95% KM (t) UCL	0.0266	95% KM (Percentile Bootstrap) UCL	0.0269
95% KM (z) UCL	0.0261	95% KM Bootstrap t UCL	0.0593
90% KM Chebyshev UCL	0.0347	95% KM Chebyshev UCL	0.0433
97.5% KM Chebyshev UCL	0.0553	99% KM Chebyshev UCL	0.0788

Gamma GOF Tests on Detected Observations Only

A-D Test Statistic	0.523	Anderson-Darling GOF Test	
5% A-D Critical Value	0.685	Detected data appear Gamma Distributed at 5% Significance Level	
K-S Test Statistic	0.328	Kolmogorov-Smirnov GOF	
5% K-S Critical Value	0.361	Detected data appear Gamma Distributed at 5% Significance Level	

Detected data appear Gamma Distributed at 5% Significance Level

Gamma Statistics on Detected Data Only

k hat (MLE)	1.858	k star (bias corrected MLE)	0.877
Theta hat (MLE)	0.00909	Theta star (bias corrected MLE)	0.0193
nu hat (MLE)	18.58	nu star (bias corrected)	8.765
Mean (detects)	0.0169		

Gamma ROS Statistics using Imputed Non-Detects

GROS may not be used when data set has > 50% NDs with many tied observations at multiple DLs

GROS may not be used when kstar of detects is small such as <1.0, especially when the sample size is small (e.g., <15-20)

For such situations, GROS method may yield incorrect values of UCLs and BTVs

This is especially true when the sample size is small.

For gamma distributed detected data, BTVs and UCLs may be computed using gamma distribution on KM estimates

Minimum	0.0058	Mean	0.0177
Maximum	0.0478	Median	0.0117
SD	0.0123	CV	0.693
Copper			
k hat (MLE)	2.884	k star (bias corrected MLE)	2.521
Theta hat (MLE)	0.00615	Theta star (bias corrected MLE)	0.00704
nu hat (MLE)	126.9	nu star (bias corrected)	110.9
Adjusted Level of Significance (β)	0.0386		
Approximate Chi Square Value (110.93, α)	87.61	Adjusted Chi Square Value (110.93, β)	86.07
95% Gamma Approximate UCL (use when n>=50)	0.0225	95% Gamma Adjusted UCL (use when n<50)	0.0229
Estimates of Gamma Parameters using KM Estimates			
Mean (KM)	0.0157	SD (KM)	0.0138
Variance (KM)	1.9140E-4	SE of Mean (KM)	0.00635
k hat (KM)	1.282	k star (KM)	1.138
nu hat (KM)	56.42	nu star (KM)	50.06
theta hat (KM)	0.0122	theta star (KM)	0.0138
80% gamma percentile (KM)	0.0249	90% gamma percentile (KM)	0.0349
95% gamma percentile (KM)	0.0449	99% gamma percentile (KM)	0.0677

Gamma Kaplan-Meier (KM) Statistics

Approximate Chi Square Value (50.06, α)	34.82	Adjusted Chi Square Value (50.06, β)	33.87
95% Gamma Approximate KM-UCL (use when n>=50)	0.0225	95% Gamma Adjusted KM-UCL (use when n<50)	0.0232

Lognormal GOF Test on Detected Observations Only

Copper

Shapiro Wilk Test Statistic	0.897	Shapiro Wilk GOF Test	
5% Shapiro Wilk Critical Value	0.762	Detected Data appear Lognormal at 5% Significance Level	
Lilliefors Test Statistic	0.277	Lilliefors GOF Test	
5% Lilliefors Critical Value	0.343	Detected Data appear Lognormal at 5% Significance Level	
Detected Data appear Lognormal at 5% Significance Level			
Lognormal ROS Statistics Using Imputed Non-Detects			
Mean in Original Scale	0.0154	Mean in Log Scale	-4.439
SD in Original Scale	0.0123	SD in Log Scale	0.733
95% t UCL (assumes normality of ROS data)	0.0199	95% Percentile Bootstrap UCL	0.0198
95% BCA Bootstrap UCL	0.0207	95% Bootstrap t UCL	0.0218
95% H-UCL (Log ROS)	0.0221		

Statistics using KM estimates on Logged Data and Assuming Lognormal Distribution

KM Mean (logged)	-4.428	KM Geo Mean	0.0119
KM SD (logged)	0.674	95% Critical H Value (KM-Log)	2.174
KM Standard Error of Mean (logged)	0.315	95% H-UCL (KM -Log)	0.0206
KM SD (logged)	0.674	95% Critical H Value (KM-Log)	2.174
KM Standard Error of Mean (logged)	0.315		

DL/2 Statistics

DL/2 Normal		DL/2 Log-Transformed	
Mean in Original Scale	0.0227	Mean in Log Scale	-3.868
SD in Original Scale	0.00819	SD in Log Scale	0.459
Copper			
95% t UCL (Assumes normality)	0.0257	95% H-Stat UCL	0.0283

DL/2 is not a recommended method, provided for comparisons and historical reasons

Nonparametric Distribution Free UCL Statistics
Detected Data appear Gamma Distributed at 5% Significance Level
Suggested UCL to Use
Copper

Gamma Adjusted KM-UCL (use when k<=1 and 15 < n < 5	0.0232

Note: Suggestions regarding the selection of a 95% UCL are provided to help the user to select the most appropriate 95% UCL.
Recommendations are based upon data size, data distribution, and skewness.
These recommendations are based upon the results of the simulation studies summarized in Singh, Maichle, and Lee (2006).
However, simulations results will not cover all Real World data sets; for additional insight the user may want to consult a statistician.

Selenium

General Statistics

Total Number of Observations	22	Number of Distinct Observations	20
Number of Detects	17	Number of Non-Detects	5
Number of Distinct Detects	17	Number of Distinct Non-Detects	3
Minimum Detect	0.147	Minimum Non-Detect	0.2
Maximum Detect	0.465	Maximum Non-Detect	0.4
Variance Detects	0.0105	Percent Non-Detects	22.73%
Mean Detects	0.299	SD Detects	0.102
Median Detects	0.317	CV Detects	0.342
Skewness Detects	-0.185	Kurtosis Detects	-1.084
Mean of Logged Detects	-1.27	SD of Logged Detects	0.384

Normal GOF Test on Detects Only

Shapiro Wilk Test Statistic	0.932	Shapiro Wilk GOF Test	
5% Shapiro Wilk Critical Value	0.892	Detected Data appear Normal at 5% Significance Level	
Lilliefors Test Statistic	0.141	Lilliefors GOF Test	
5% Lilliefors Critical Value	0.207	Detected Data appear Normal at 5% Significance Level	

Detected Data appear Normal at 5% Significance Level

Kaplan-Meier (KM) Statistics using Normal Critical Values and other Nonparametric UCLs

KM Mean	0.278	KM Standard Error of Mean	0.0236
KM SD	0.102	95% KM (BCA) UCL	0.316
95% KM (t) UCL	0.319	95% KM (Percentile Bootstrap) UCL	0.318
95% KM (z) UCL	0.317	95% KM Bootstrap t UCL	0.317
90% KM Chebyshev UCL	0.349	95% KM Chebyshev UCL	0.381
97.5% KM Chebyshev UCL	0.426	99% KM Chebyshev UCL	0.513

Gamma GOF Tests on Detected Observations Only

A-D Test Statistic	0.672	Anderson-Darling GOF Test	
5% A-D Critical Value	0.74	Detected data appear Gamma Distributed at 5% Significance Level	
K-S Test Statistic	0.159	Kolmogorov-Smirnov GOF	
5% K-S Critical Value	0.209	Detected data appear Gamma Distributed at 5% Significance Level	

Detected data appear Gamma Distributed at 5% Significance Level

Gamma Statistics on Detected Data Only

k hat (MLE)	7.98	k star (bias corrected MLE)	6.611
Theta hat (MLE)	0.0375	Theta star (bias corrected MLE)	0.0453
nu hat (MLE)	271.3	nu star (bias corrected)	224.8
Mean (detects)	0.299		

Gamma ROS Statistics using Imputed Non-Detects

GROS may not be used when data set has > 50% NDs with many tied observations at multiple DLs

GROS may not be used when kstar of detects is small such as <1.0, especially when the sample size is small (e.g., <15-20)

For such situations, GROS method may yield incorrect values of UCLs and BTVs

This is especially true when the sample size is small.

For gamma distributed detected data, BTVs and UCLs may be computed using gamma distribution on KM estimates

Minimum	0.147	Mean	0.281
Maximum	0.465	Median	0.283
SD	0.0983	CV	0.35
k hat (MLE)	8.211	k star (bias corrected MLE)	7.121
Selenium			
Theta hat (MLE)	0.0342	Theta star (bias corrected MLE)	0.0395
nu hat (MLE)	361.3	nu star (bias corrected)	313.3
Adjusted Level of Significance (β)	0.0386		
Approximate Chi Square Value (313.33, α)	273.3	Adjusted Chi Square Value (313.33, β)	270.5
95% Gamma Approximate UCL (use when n>=50)	0.322	95% Gamma Adjusted UCL (use when n<50)	0.325
Estimates of Gamma Parameters using KM Estimates			
Mean (KM)	0.278	SD (KM)	0.102
Variance (KM)	0.0105	SE of Mean (KM)	0.0236
k hat (KM)	7.389	k star (KM)	6.412
nu hat (KM)	325.1	nu star (KM)	282.1
theta hat (KM)	0.0377	theta star (KM)	0.0434
80% gamma percentile (KM)	0.364	90% gamma percentile (KM)	0.425
95% gamma percentile (KM)	0.48	99% gamma percentile (KM)	0.596

Gamma Kaplan-Meier (KM) Statistics

Approximate Chi Square Value (282.12, α)	244.2	Adjusted Chi Square Value (282.12, β)	241.6
95% Gamma Approximate KM-UCL (use when n>=50)	0.322	95% Gamma Adjusted KM-UCL (use when n<50)	0.325

Lognormal GOF Test on Detected Observations Only

Selenium

Shapiro Wilk Test Statistic	0.889	Shapiro Wilk GOF Test	
5% Shapiro Wilk Critical Value	0.892	Detected Data Not Lognormal at 5% Significance Level	
Lilliefors Test Statistic	0.172	Lilliefors GOF Test	
5% Lilliefors Critical Value	0.207	Detected Data appear Lognormal at 5% Significance Level	
Detected Data appear Approximate Lognormal at 5% Significance Level			
Lognormal ROS Statistics Using Imputed Non-Detects			
Mean in Original Scale	0.279	Mean in Log Scale	-1.339
SD in Original Scale	0.0992	SD in Log Scale	0.372
95% t UCL (assumes normality of ROS data)	0.316	95% Percentile Bootstrap UCL	0.313
95% BCA Bootstrap UCL	0.317	95% Bootstrap t UCL	0.317
95% H-UCL (Log ROS)	0.328		

Statistics using KM estimates on Logged Data and Assuming Lognormal Distribution			
KM Mean (logged)	-1.353	KM Geo Mean	0.259
KM SD (logged)	0.394	95% Critical H Value (KM-Log)	1.907
KM Standard Error of Mean (logged)	0.0915	95% H-UCL (KM -Log)	0.329
KM SD (logged)	0.394	95% Critical H Value (KM-Log)	1.907
KM Standard Error of Mean (logged)	0.0915		

DL/2 Statistics			
DL/2 Normal		DL/2 Log-Transformed	
Mean in Original Scale	0.266	Mean in Log Scale	-1.423
SD in Original Scale	0.112	SD in Log Scale	0.468
95% t UCL (Assumes normality)	0.307	95% H-Stat UCL	0.329
DL/2 is not a recommended method, provided for comparisons and historical reasons			

Nonparametric Distribution Free UCL Statistics	
Detected Data appear Normal Distributed at 5% Significance Level	
Suggested UCL to Use	
95% KM (t) UCL	0.319

Note: Suggestions regarding the selection of a 95% UCL are provided to help the user to select the most appropriate 95% UCL.
Recommendations are based upon data size, data distribution, and skewness.
These recommendations are based upon the results of the simulation studies summarized in Singh, Maichle, and Lee (2006).
However, simulations results will not cover all Real World data sets; for additional insight the user may want to consult a statistician.

Zinc

General Statistics

Total Number of Observations	22	Number of Distinct Observations	6
Number of Detects	4	Number of Non-Detects	18
Number of Distinct Detects	4	Number of Distinct Non-Detects	2
Minimum Detect	0.0307	Minimum Non-Detect	0.01
Maximum Detect	0.356	Maximum Non-Detect	0.1
Variance Detects	0.0207	Percent Non-Detects	81.82%
Mean Detects	0.157	SD Detects	0.144
Median Detects	0.121	CV Detects	0.914
Skewness Detects	1.183	Kurtosis Detects	0.954
Mean of Logged Detects	-2.22	SD of Logged Detects	1.05

Normal GOF Test on Detects Only

Shapiro Wilk Test Statistic	0.916	Shapiro Wilk GOF Test	
5% Shapiro Wilk Critical Value	0.748	Detected Data appear Normal at 5% Significance Level	
Lilliefors Test Statistic	0.226	Lilliefors GOF Test	
5% Lilliefors Critical Value	0.375	Detected Data appear Normal at 5% Significance Level	
Detected Data appear Normal at 5% Significance Level			

Kaplan-Meier (KM) Statistics using Normal Critical Values and other Nonparametric UCLs

KM Mean	0.0593	KM Standard Error of Mean	0.0241
KM SD	0.0748	95% KM (BCA) UCL	N/A
95% KM (t) UCL	0.101	95% KM (Percentile Bootstrap) UCL	N/A
95% KM (z) UCL	0.099	95% KM Bootstrap t UCL	N/A
90% KM Chebyshev UCL	0.132	95% KM Chebyshev UCL	0.164
97.5% KM Chebyshev UCL	0.21	99% KM Chebyshev UCL	0.299

Gamma GOF Tests on Detected Observations Only

A-D Test Statistic	0.204	Anderson-Darling GOF Test	
5% A-D Critical Value	0.662	Detected data appear Gamma Distributed at 5% Significance Level	
K-S Test Statistic	0.19	Kolmogorov-Smirnov GOF	
5% K-S Critical Value	0.4	Detected data appear Gamma Distributed at 5% Significance Level	
Detected data appear Gamma Distributed at 5% Significance Level			

Gamma Statistics on Detected Data Only

k hat (MLE)	1.494	k star (bias corrected MLE)	0.54
Theta hat (MLE)	0.105	Theta star (bias corrected MLE)	0.291
nu hat (MLE)	11.95	nu star (bias corrected)	4.321
Mean (detects)	0.157		

Gamma ROS Statistics using Imputed Non-Detects

GROS may not be used when data set has > 50% NDs with many tied observations at multiple DLs

GROS may not be used when kstar of detects is small such as <1.0, especially when the sample size is small (e.g., <15-20)

For such situations, GROS method may yield incorrect values of UCLs and BTVs

This is especially true when the sample size is small.

For gamma distributed detected data, BTVs and UCLs may be computed using gamma distribution on KM estimates

Minimum	0.01	Mean	0.0596
Maximum	0.356	Median	0.0222
SD	0.082	CV	1.376
Zinc			
k hat (MLE)	0.833	k star (bias corrected MLE)	0.749
Theta hat (MLE)	0.0716	Theta star (bias corrected MLE)	0.0796
nu hat (MLE)	36.63	nu star (bias corrected)	32.97
Adjusted Level of Significance (β)	0.0386		
Approximate Chi Square Value (32.97, α)	20.84	Adjusted Chi Square Value (32.97, β)	20.12
95% Gamma Approximate UCL (use when n>=50)	0.0943	95% Gamma Adjusted UCL (use when n<50)	N/A
Estimates of Gamma Parameters using KM Estimates			
Mean (KM)	0.0593	SD (KM)	0.0748
Variance (KM)	0.0056	SE of Mean (KM)	0.0241
k hat (KM)	0.629	k star (KM)	0.573
nu hat (KM)	27.66	nu star (KM)	25.22
theta hat (KM)	0.0943	theta star (KM)	0.103
80% gamma percentile (KM)	0.0977	90% gamma percentile (KM)	0.156
95% gamma percentile (KM)	0.217	99% gamma percentile (KM)	0.365

Gamma Kaplan-Meier (KM) Statistics

Approximate Chi Square Value (25.22, α)	14.78	Adjusted Chi Square Value (25.22, β)	14.18
95% Gamma Approximate KM-UCL (use when n>=50)	0.101	95% Gamma Adjusted KM-UCL (use when n<50)	0.105

Lognormal GOF Test on Detected Observations Only

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Shapiro Wilk Test Statistic	0.995	Shapiro Wilk GOF Test	
5% Shapiro Wilk Critical Value	0.748	Detected Data appear Lognormal at 5% Significance Level	
Lilliefors Test Statistic	0.157	Lilliefors GOF Test	
5% Lilliefors Critical Value	0.375	Detected Data appear Lognormal at 5% Significance Level	
Detected Data appear Lognormal at 5% Significance Level			
Lognormal ROS Statistics Using Imputed Non-Detects			
Mean in Original Scale	0.0594	Mean in Log Scale	-3.39
SD in Original Scale	0.0777	SD in Log Scale	1.08
95% t UCL (assumes normality of ROS data)	0.0879	95% Percentile Bootstrap UCL	0.0886
95% BCA Bootstrap UCL	0.101	95% Bootstrap t UCL	0.115
95% H-UCL (Log ROS)	0.114		

Statistics using KM estimates on Logged Data and Assuming Lognormal Distribution

KM Mean (logged)	-3.358	KM Geo Mean	0.0348
KM SD (logged)	1.011	95% Critical H Value (KM-Log)	2.598
KM Standard Error of Mean (logged)	0.528	95% H-UCL (KM -Log)	0.103
KM SD (logged)	1.011	95% Critical H Value (KM-Log)	2.598
KM Standard Error of Mean (logged)	0.528		

DL/2 Statistics

DL/2 Normal		DL/2 Log-Transformed	
Mean in Original Scale	0.0675	Mean in Log Scale	-2.959
SD in Original Scale	0.0702	SD in Log Scale	0.723
95% t UCL (Assumes normality)	0.0932	95% H-Stat UCL	0.0958

DL/2 is not a recommended method, provided for comparisons and historical reasons

Nonparametric Distribution Free UCL Statistics
Detected Data appear Normal Distributed at 5% Significance Level

Suggested UCL to Use

95% KM (t) UCL	0.101

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Note: Suggestions regarding the selection of a 95% UCL are provided to help the user to select the most appropriate 95% UCL.
Recommendations are based upon data size, data distribution, and skewness.
These recommendations are based upon the results of the simulation studies summarized in Singh, Maichle, and Lee (2006).
However, simulations results will not cover all Real World data sets; for additional insight the user may want to consult a statistician.